

# OPTIMIZATION OF MANUFACTURING PARAMETERS FOR ASHMOH CEMENT

by

HEMENDRA KUMAR GUPTA

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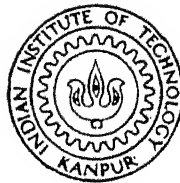
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DEPARTMENT OF METALLURGICAL ENGINEERING  
INDIAN INSTITUTE OF TECHNOLOGY KANPUR

AUGUST, 1978

# **OPTIMIZATION OF MANUFACTURING PARAMETERS FOR ASHMOH CEMENT**

**A Thesis Submitted  
In Partial Fulfilment of the Requirements  
for the Degree of  
MASTER OF TECHNOLOGY**

**by  
HEMENDRA KUMAR GUPTA**

**to the**

**DEPARTMENT OF METALLURGICAL ENGINEERING  
INDIAN INSTITUTE OF TECHNOLOGY KANPUR  
AUGUST, 1978**

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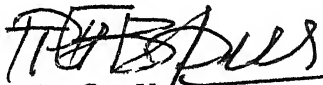
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C E R T I F I C A T E

This is to certify that this work entitled  
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by H.K. Gupta has been carried out under my supervision  
and has not been submitted elsewhere for a degree.

Kanpur  
August 7 1978

  
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### ABSTRACT

Samples of ASHMOH were prepared using different grinding media and varying the grinding time. Apparent density, tap density and specific surface area of the cement and Tensile Strength and Bulk density of cement - sand mortar samples were determined. It was found that optimal grinding time was 8 hours. The most effective grinding media were found to be large cylinders for the 8 hours optimum grinding time.

Portland replacement in ASHMOH enhances its Tensile strength. When interground with ASHMOH, 10 per cent portland replacement is found optimum, providing strength properties comparable to that of portland cement.

RHA-PORT cement, a new cement prepared by mixing portland cement with rice husk ash, ground for 8 hours, using large cylindrical grinding media, in 1:1 ratio, gives comparable strength values with portland cement and has substantially higher resistance to chemical corrosion.



## CHAPTER 1

### INTRODUCTION

On fine grinding a dry mixture of rice husk ash and lime in appropriate proportions in a ball mill we obtain a hydraulic setting cement called Ashmoh cement. The process of making Ashmoh cement was developed at I.I.T. Kanpur. Ashmoh is being made on pilot plant scale in the district Banda of U.P. with encouraging results. Laboratory tests show that cementitious material can compete with portland cement in masonry works and the manufacture of cement blocks, tiles and precast-irrigation channels. Ashmoh cement is more resistant to chemical corrosion than portland cement because of its high silica and low lime content.

There is a wide gap between demand and supply of cement in India and this gap is increasing day by day. Cement also has tremendous export potential to South-East Asia and Gulf countries. Hence it can become an important source of valuable foreign exchange. This amply demonstrates the need for increasing the production of cement. However setting up new portland cement plants entails various problems.

(1) The raw material is available only at selective locations in the country and, therefore, the plant has to be set up near such locations which are usually far away from the major centres of consumption.

(2) Complete infrastructure for the plant has to be developed if it is located away from the industrial centres. This naturally increases the capital and operating costs.

(3) Finally continuous supply of suitable fuel for cement kilns is becoming increasingly problematic because of overall squeeze on energy sources.

Hence setting up of new cement plant require massive investment and fairly large gestation time. Moreover, this industry has a limited employment potential. Clearly there is a need for a labour intensive appropriate technology to manufacture building materials including cement, which can consume local raw material and which in turn can be consumed locally.

Ashmoh cement is an appropriate alternative cementing material. It utilizes rice husk ash as the major raw material. Rice husk ash is obtained as waste material in rice mills after burning the husk in boilers. But the quantity of rice husk ash available in a particular region limits the size of the Ashmoh cement plant, which is not

necessarily a disadvantage from the point of view of setting up decentralized production units.

Ashmoh has been tested for its cementing properties such as strength, initial and final setting time. However, no work had been done so far to optimize the process variables in order to improve its commercial viability. The main objective of the present work is to optimize the various manufacturing parameters such as grinding time, charge weight, shape of the grinding media and amount of a suitable additive. This is necessary because the grinding operation in the manufacture of Ashmoh contributes substantially to the overall cost of production. Furthermore, an attempt has also been made to produce a new variety of cement by blending finely ground ash with portland cement, which gives required strength values but is superior in corrosion resistance to organic and inorganic acids than ordinary portland cement.

## CHAPTER 2

### LITERATURE REVIEW

2.1 Pozzolanas- are either materials which occur naturally e.g. volcanic deposits or diatomaceous earth or are derived artificially as burnt clays, shales and gaize or fly ash. Eventhough not cementitious in themselves, pozzolanas contain constituents which in a finely divided state combine with lime, in the presence of water, to form stable insoluble compounds possessing cementitious properties. The reactive constituent of pozzolan is primarily silica ( $\text{SiO}_2$ ) and to a lesser extent Alumina ( $\text{Al}_2\text{O}_3$ ) or  $\text{Fe}_2\text{O}_3$ . These oxides have to be present in a reactive form, glassy or <sup>p</sup>crystalline phase, in order to combine with lime to form complex compounds which render cementitious properties to pozzolanic cement.

### 2.2 Chemical Composition of Pozzolanas:

A pozzolan as per ASTM C 618 contains 70 per cent or more of  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ . Chemical specification, however, is of little significance in determining the cement potential. In general a high percentage of amorphous silica content is essential. Composition of various artificial and natural pozzolanas is given in tables 2.1 and 2.2. It is evident that pozzolanas exhibit a wide range of chemical compositions. Pozzolanas

Table 2.1 Percentage composition of volcanic ash-natural pozzolanas (12)

Pozzolanas	Ignition loss	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	SO <sub>3</sub>
Rhenish Trass	10.1	54.6	16.4	3.8	0.6	3.8	1.9	5.1	3.9	0.4
"	8.5	54.8	17.2	4.4	0.6	2.3	0.9	7.0	3.8	0.1
Bavarian Trass	14.5	57.0	10.9	5.6	0.5	6.0	2.2	1.8	1.5	0.2
Santorin earth	4.9	63.2	13.2	4.9	1.0	4.0	2.1	3.9	2.6	0.7
"	3.1	65.2	12.9	6.3	-	3.2	1.9	2.6	4.2	-
Rome: Segni	9.6	44.1	17.3	10.7	-	12.0	2.0	1.4	3.1	-
Segni	5.3	48.2	21.9	9.6	-	7.5	3.2	(4.1)		0.3
S.Paolo	4.1	45.2	20.0	10.7	-	9.8	3.8	(6.2)		0.3
Naples: Baroli	4.8	55.7	19.0	4.6	-	5.0	1.3	3.4	3.9	-
Baia	4.4	59.5	19.3	3.3	-	2.1	0.2	(11.3)		0.2
Rumanian Trass	13.9	62.5	11.6	1.8	-	6.6	0.7		2.9	-
Crimean tuff	11.7	70.1	10.7	1.0	-	2.5	0.3		3.7	-
USA rhyolitic	3.4	65.7	15.9	2.5	-	3.4	1.3	5.0	1.9	-
Pumicite	4.2	72.3	13.3	1.4	-	0.7	0.4	1.6	5.6	Traces

Table 2.2 Percentage composition of some artificial pozzolanas (16)

Pozzolana	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O or K <sub>2</sub> O	SO <sub>3</sub>	Ignition loss
Burnt clay	60.2	17.7	7.6	2.7	2.5	4.2	2.5	1.3
Spent oil shale	51.7	22.4	11.2	4.3	1.1	3.6	2.1	3.2
Burnt gaize	88.0	6.4	3.3	1.2	0.8	-	Trace	-
Raw moler	66.7	11.4	7.8	2.2	2.1	-	1.4	5.6
Burnt moler	70.7	12.1	8.2	2.3	2.2	-	1.5	-
Raw dolomite	86.7	2.3	1.8	Trace	0.6	0.4	-	8.3
Burnt dolomite	69.7	14.7	8.1	1.5	2.2	3.2	-	0.4
Fly ash (USA)	47.1	18.2	19.2	7.0	1.1	3.95	2.8	1.2
Fly ash (British)	47.4	27.5	10.3	2.1	2.0	5.7	1.8	0.9

are used either as admixture with portland cement or as lime-pozzolana cements. Our primary interest in this investigation is in the second effect.

### 2.3 PROPERTIES OF LIME-POZZOLANA MORTARS:

Lime-pozzolana cements and mortars are characterized by

- (i) High chemical resistance
- (ii) High cracking resistance
- (iii) Medium to high dry shrinkage
- (iv) Low to medium rate of evolution of heat of hydration
- (v) Low rate of strength development

The setting time is variable and though the initial set may occur in 1-3 hours the final set usually occurs in 10-12 hours.

The strength developed in lime pozzolana mortars varies with the ratio of lime to pozzolana in the mix. At early ages the maximum strength is obtained with a lime-pozzolana ratio of about 1:4 with finely ground materials, but at longer ages the optimum ratio moves towards mixes of higher lime content and approaches about 1:3 to 1:2 at one year (Table 2.4). The rate of development of strength is increased by rise in

Table 2.3 (12)

Pozzolana	Maximum strength (kg/cm <sup>2</sup> )		Percentage of lime (in the lime - pozzolana mix) giving maximum strength	
	Bending	Compression	Bending	Compression
Trass	38.6	115	35	26
Roman	35.5	130	32	28

Table 2.4 Effect of pozzolana : lime ratio on strength of mortars (12)

Pozzolana	Mix proportions (weight)			Tensile strength (kg/cm <sup>2</sup> )			
	Hydrated lime	Pozzolana	Standard sand	7 days	28 days	90 days	1 year
Burnt shale	1	1	6	7.51	14.55	24.0	36.5
	1	2	9	9.34	22.6	32.3	39.4
	1	4	15	14.28	26.1	36.1	37.5
Trass	1	1	6	15.0	25.4	31.4	33.6
	1	2	9	15.8	27.4	29.9	34.8
	1	4	15	16.45	25.5	29.0	30.4



Table 2.5 Tests on 0.8:1:1.5 hydrated lime : pozzolana: standard sand mortars (12)

Pozzolana	Percentage residue on 170 - mesh sieve	Tensile strength (kg/cm <sup>2</sup> )	Compressive strength (kg/cm <sup>2</sup> )
		7 days	28 days
Italian	33.8	15.05	25.0
Trass	35.5	8.43	17.9
Moler	49.2	12.50	18.95
Punice	29.2	5.06	18.95
			34.8
			104.0

Table 2.6 Effect of fineness of grinding of pozzolanas 1:4:15 lime:trass:graded sand mortars (14.8 percent water) (12)

Percent residue on 170 mesh sieve	Strength		
	Bending	Compression	
	28 days	28 days	1 year
43.0	12.85	24.0	83.5
14.5	18.9	35.1	89.9
3.0	21.5	48.5	102.5

Table 2.7 Effect of temperature on strength of lime - pozzolana mortars (1:1:6 lime : pozzolana : standard mortars of dry consistence) (12)

Pozzolana	Tensile Strength			
	7 days	25°	35°	0°
	12°	25°	35°	0°
Santorin earth	0	0.562	3.09	7.24
Burnt shale	3.375	6.04	10.6	18.0
			6.46	16.92
			24.45	28.7
			32.0	

temperature. Pozzolana that is inert at low temperatures ( $15^{\circ}\text{C}$ ) may show satisfactory results at higher temperatures ( $40^{\circ}\text{C}$ ) (Table 2.7). Additives can give rise to favourable effects as regards setting time and strength development in shorter time period<sup>(12)</sup>.

Pozzolana-lime mortars attain a much higher ultimate strength when cured in water than in air, though the initial effect is normally the reverse. With lime pozzolana concretes in particular a long period of moist curing is essential to the development of high strengths. Rapid drying has a most injurious effect<sup>(12)</sup>.

From table 2.3-2.7 we conclude the

- (i) effect of lime content
- (ii) optimal lime content
- (iii) effect of fineness
- (iv) effect of the temperature.

#### 2.4 MECHANISM OF LIME-POZZOLANA REACTION:

The fundamental property of a pozzolana is its ability to combine with lime. Direct combination theory has been able to explain this phenomenon satisfactorily. According to this theory, hydrated lime reacts with pozzolana in presence of water to form calcium silicate hydrates. X-ray work carried out by Malquori,<sup>(25)</sup> Lea and<sup>(26)</sup> Stratting<sup>(27)</sup> found the presence of  $4 \text{ CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{aq.}$ ,

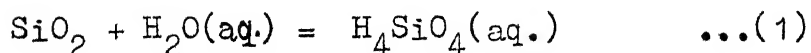
of  $3\text{CaO} \cdot \text{SiO}_2$  with  $\text{CaO} \cdot \text{SiO}_2$  aq. in the hydrated lime pozzolana mix.

Hydrothermal reaction of lime with silica has been studied extensively for the manufacture of hydrated calcium silicate autoclaved products. It is expected that in the case of lime-silica reactive pozzolanas also the reaction mechanism will be similar although the kinetics would be retarded under the conditions of ambient temperature curing.

It is postulated that the reaction mechanism proceeds as follows:

(1) Chemisorption of calcium hydroxide by surface silanol groups. Amount of calcium hydroxide chemisorbed can be correlated with the number of silanol groups on the surface.

(2) The solution of silica in the aqueous phase. Silica reacts with water to form a saturated solution of monosilicic acid. The solubility of the increases with pH because of the form-



ation of  $\text{H}_3\text{SiO}_4^-$  and  $\text{H}_2\text{SiO}_4^{2-}$  ions<sup>3</sup>. Silica has been reported<sup>(4)</sup> to dissolve in sodium hydroxide solutions (to form a monosilicic acid solution) as a function of the surface area  $S$  of the silica

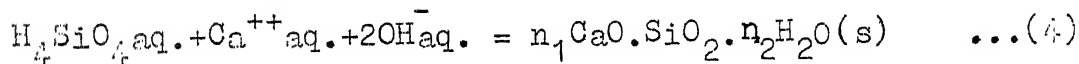
$$\frac{dc}{dt} = K_1 S \quad \dots(2)$$

Where  $C$  is the concentration of monosilicic acid in moles/L. and  $K_1$  is the rate constant with the assumption that the silica particles are spherical and monodisperse, the integrated form of Eq. 2 may be derived

$$C_p^{1/3} = C_{p0}^{1/3} - K_2 t \quad \dots(3)$$

where  $C_p$  and  $C_{p0}$  are proportional to the amount of undissolved silica at time  $t$  and zero time, respectively, and  $K_2$  is a constant.

(3) The reaction of monosilicic acid or its ions in solution with calcium hydroxide in solution.



(4) Formation of nuclei of calcium silicate hydrate.

(5) Growth of nuclei

(6) Flocculation and precipitation of crystals.

The velocity of any of these processes might be rate determining. However step 2, the solution of silica controls the kinetics of the overall reaction<sup>(5)</sup>.

In India, natural pozzolanas of volcanic origin are not available. Other primary pozzolanas like pumice are very expensive. Artificial cheap pozzolana like fly ash is not reactive enough to combine with lime and give proper cementitious material. India is a major rice producing country with 50 MT paddy production/annum.

## 2.5 RICE HUSK ASH:

Rice husk is a waste product in rice mills after burning the rice husk in boilers. Burning operation produces large quantities of ash, about 20 per cent by weight of husks. India produces around 2 MT of rice husk ash per annum.

Rice husk, completely burnt, is light grey in colour, of very high surface area and is very friable. It is a possible pozzolanic material as it is highly siliceous. As can be seen from the table <sup>2.8</sup> / its silica content is approximately 90 per cent and carbon content is variable depending on the combustion condition but usually below 7 per cent. Previous works have reported that silica is present in highly porous, distorted multiple polymorphic forms with an enormous internal surface area. Part of the silica is found to be semiamorphous. Consequently this ash is of very low bulk density and is highly reactive.

The degree of crystallinity varies with temperature and time of the combustion of husk. Lower temperature and time yields relatively high amorphous silica content. With increased time and temperature crystalline forms of silica namely cristobalite and strangely enough quartz increases and amorphous silica decreases (Fig. 2.1)<sup>(7)</sup>. Boiler and field burning gives

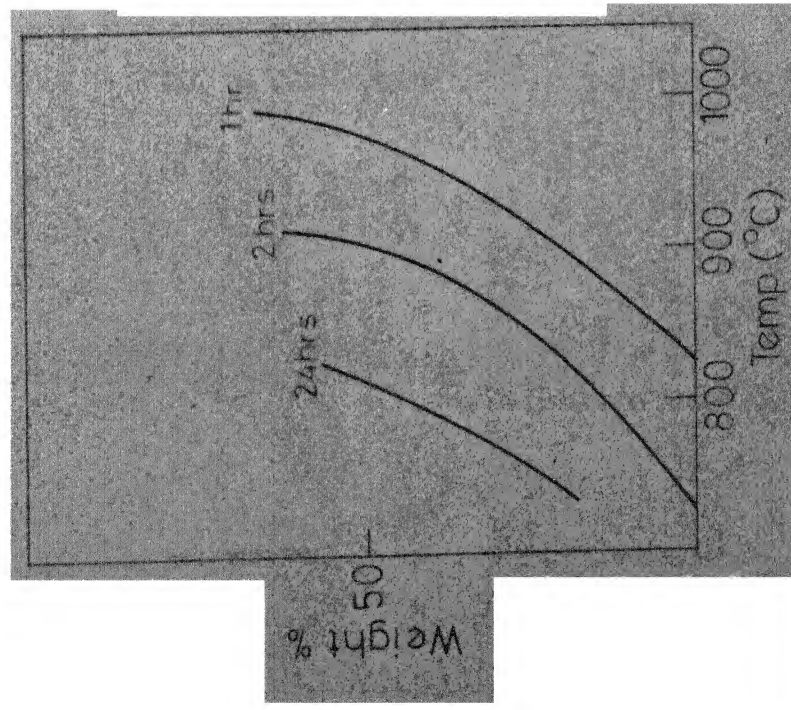


Fig 2 1 - Crystallization of cristobolite with variation of temperature and time.

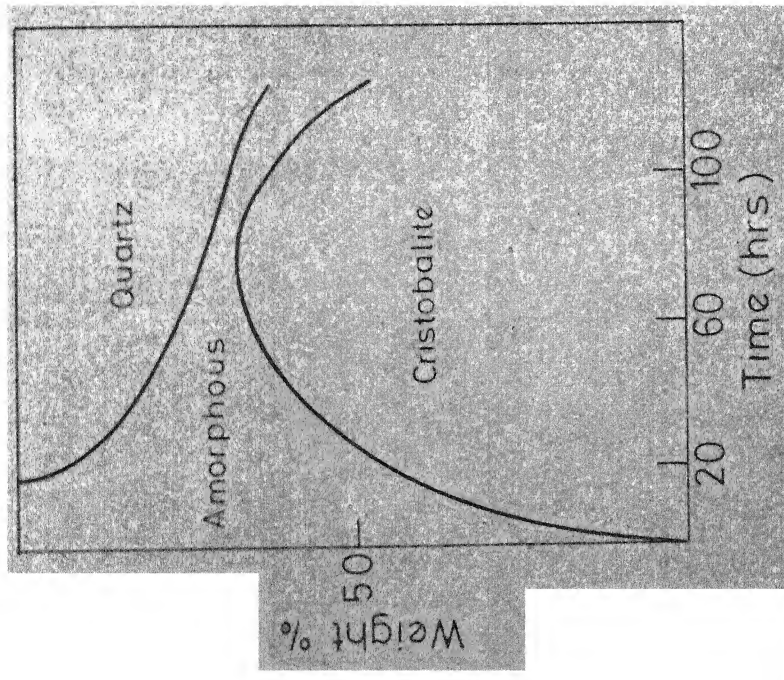


Fig 2 2 - Crystallization of cristobolite and quartz at 800 °C with variation of time.

Table 2.8. Chemical Analysis of RHA

Constituent	Ash No. 1 (14)	Ash No. 2 (14)	Ash No. 3 (18)
SiO <sub>2</sub>	78.75	86.10	94.50
Fe <sub>2</sub> O <sub>3</sub> + Al <sub>2</sub> O <sub>3</sub> + TiO <sub>2</sub>	6.40	3.70	Traces
Fe <sub>2</sub> O <sub>3</sub>	1.95	1.60	-
CaO	0.70	1.10	0.25
MgO	1.42	0.90	0.23
Na <sub>2</sub> O + K <sub>2</sub> O	10.26	5.52	1.88
Loss of ignition	2.51	2.68	-
P <sub>2</sub> O <sub>3</sub>	-	-	0.53
SO <sub>3</sub>	-	-	1.13

relatively crystalline silica.

Various characteristics of rice husk ash makes it a very unique material which can be usefully exploited. So far it has found use in making porous refractories<sup>(17)</sup>, fillers for plastics<sup>(19)</sup>, lining for ingot moulds<sup>(20)</sup>, acoustical boards<sup>(21)</sup>, porous media<sup>(22)</sup>, Silicon carbide<sup>(23)</sup>, silicon tetrachloride<sup>(24)</sup>, Ashmoh Cement and light weight concrete<sup>(15)</sup>.

## 2.6 RICE HUSK ASH CEMENT:

High silica content and cheap/free availability of rice husk ash has attracted quite a few people for its exploitation. As mentioned before its similarity with pozzolanas has led to its use in building materials. Rice husk ash cement is result of extensive research works done in India and abroad. Rice husk ash mixed with lime in predetermined proportion is finely ground (determination of the optimum grinding parameters is, in fact, the main objective of this work) to produce a material with cementing properties. This is known as rice husk ash cement. Kapur and Shrivastava have established the feasibility of producing cement from rice husk ash. P.K. Mehta has also shown the potential of rice husk ash for making cement. Difference between P.K.Mehta and Kapur is that the former uses amorphous rice husk ash produced under controlled burning condition while the later uses rice husk



ash thrown out as total waste material by the rice mills. Process developed by Kapur is more suited to the Indian conditions as most of the rice mills are operating on boilers which use rice husk as the fuel.

Bajpai's work shows that boiler ash is both amorphous and crystalline. This is presumably due to the fact that the burning conditions are seldom uniform in the boiler. On grinding fresh surfaces having disturbed crystalline layers on the surface are formed which increase the reactivity of the ash. Calcium oxide present in the mixture reacts with this surface in presence of water to form nuclei of hydrated calcium silicates. This implies faster rate of strength development and depletion of lime in the cement paste. X-ray analysis done by Bajpai shows diminishing peaks of  $\text{Ca(OH)}_2$  and formation of new peaks corresponding to following compounds

- (i)  $2\text{CaO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ ,  $\alpha$  Hydrate
- (ii)  $2\text{CaO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$
- (iii)  $3\text{CaO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$
- (iv)  $\text{Ca}_4(\text{Si}_6\text{O}_{15})(\text{OH})_2 \cdot 3\text{H}_2\text{O}$
- (v)  $\text{Ca}_7(\text{CO}_3)(\text{Si}_6\text{O}_{18}) \cdot 2\text{H}_2\text{O}$
- (vi)  $6\text{CaO} \cdot 4\text{SiO}_2 \cdot 3\text{H}_2\text{O}$
- (vii)  $\text{CaCO}_3$

According to Jain a commercially produced ashmoh has low true density ( $2082.5 \text{ Kg/m}^3$ ) and bulk density ( $950 \text{ Kg/m}^3$ ) compared to portland cement. Surface

area was found to be  $3850 \text{ cm}^2/\text{g}$ . Its initial setting time is 45 minutes and final setting time is 5 hours. Strength of 1:3 Ashmoh cement - sand mortar cubes is about  $160 \text{ Kg}/\text{cm}^2$  in three days and about  $200 \text{ Kg}/\text{cm}^2$  in seven days. These values are sufficient for masonry and plastering in many structural applications and comparable to that of Portland Cement. This cement maintains its superiority over Portland cement in terms of chemical resistance and long term durability.

Like Portland cement it can be used in making prefabricated structures, ferrocement spun pipes etc. Ashmoh cement has also found use in making light weight concrete<sup>(15)</sup>.

## 2.7 GRINDING:

The purpose of grinding is to disintegrate solid particles and form the large surface area and surface active sites by supplying mechanical energy. Dempster and Ritchie have found that grinding produces a disturbed layer on the surface of siliceous particles. The external layer is highly reactive and forms calcium silicate hydrate with calcium hydroxide in water. This layer of calcium silicate hydrate travels gradually to the centre of pozzolana particles. The whole process is very slow and takes a very long time to complete.

By fine grinding we mean production of particles of size  $< 50\mu\text{m}$ . The extent of fineness attained is determined by two counteracting processes (i) increase of free surface accompanied by particle size reduction and (ii) decrease of free surface accompanied by particle size increase.

In case of particle size increase two stages can be identified. First, aggregation, i.e. weak, reversible adhesion of particles due to Vander waals forces, occurring during grinding for short times, and, second for long durations, a very compact, irreversible adhesion of the particles due to chemical forces which are stronger by several orders of magnitude, i.e. agglomeration.

Prolonged grinding not only changes the physical characteristics but chemical characteristics (polymorphic transformations, catalytic activity etc.) also. In case of dry grinding the effect on chemical properties is much more as compared to wet grinding<sup>(9)</sup>

## 2.8 GRINDING PARAMETERS AND OPTIMIZATION:

Size reduction involves a combination of grinding through impact, compression and attrition, where strain energy, shear energy, thermal energy and kinetic energy induce various changes in the state of solids. In the mechanics of ball milling, energy supplied to a particle during size reduction

would be distributed in (i) an increase in the surface energy of particles (ii) elastic deformation of the particles (iii) plastic deformation and (iv) gliding or slip on crystallographic planes, twinning and other possible lattice rearrangements, leading to amorphisation of the solid mostly in thin surface layer.

At the start of the process, when the number of flaws and pores is large, size reduction takes place relatively at a faster rate. In this stage (marked by 'a' in Fig. 2.3) the surface area produced tends to be proportional to the grinding time (Rathinger's law of grinding). With advance of the grinding, the number of small particle increases and consequently grinding resistance of material increases. Simultaneously adhesion of particles to the grinding media to the mill lining, as well as to each other begins. The power consumed for grinding is no longer proportional to the increase in surface. This stage will be called aggregation. (marked by 'b' in Fig. 2.3)

After reaching a certain fineness, the increase in degree of dispersion reaches a maximum value; moreover, for certain materials, e.g. rice husk ash and lime mix., a decrease in the degree of dispersion may appear (stage c in Fig. 2.3). This is the stage of agglomeration which is characterized by crystal structure and mechano-chemical

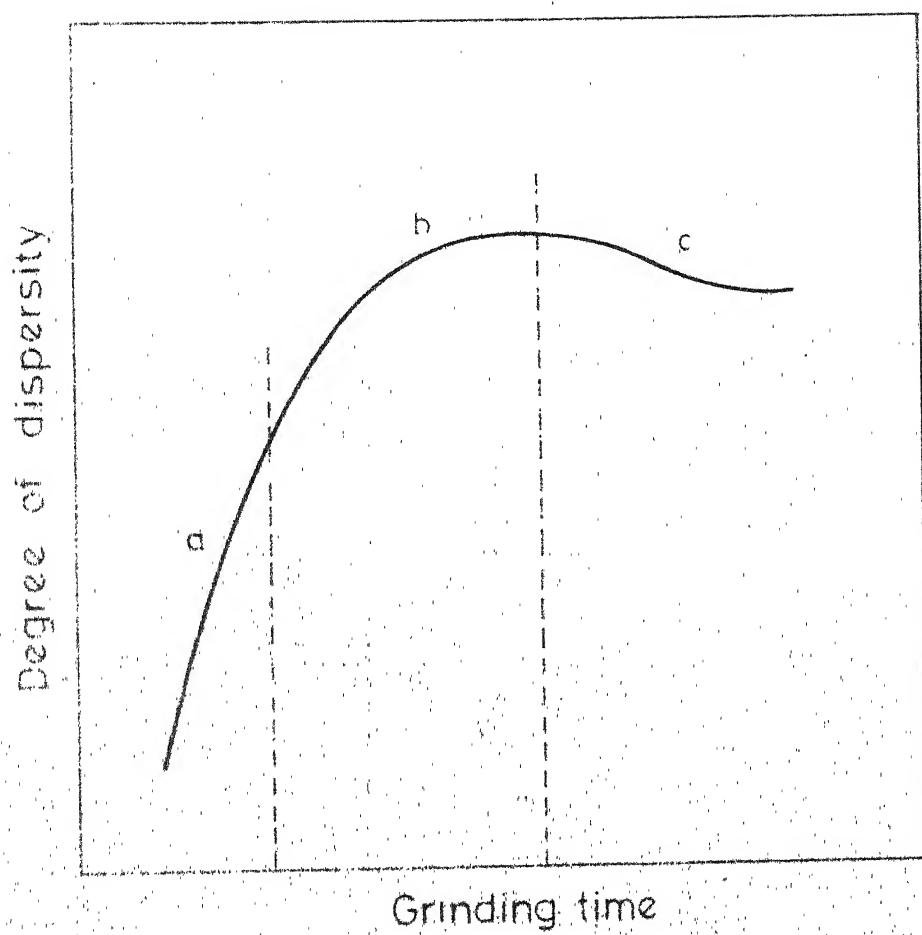


Fig 2 3

changes in the material.

Hence there is a need to optimize the grinding process in view of power consumption and production capacity. Following parameters need to be optimized to determine equilibrium conditions for grinding by ball mill.

1. Size of the mill.
2. Speed of the mill.
3. Time of milling.
4. Size of balls
5. Shape of balls
6. Material of balls
7. Amount of balls
8. Ratio of amount of material/volume of mill
9. Ratio of amount of material/amount of balls
10. Medium of milling.

In the present work optimization is done for Ashmoh Cement wrt time of milling. Size of balls, shape of balls and ratio of amount of material/amount of balls.

### CHAPTER 3

#### OBJECTIVE OF PRESENT WORK

Ashmoh is produced by a single grinding operation. The main objective of this work is to establish the optimal grinding parameters for a ball mill. A new type of cement has also been tried by blending finely ground rice husk ash with portland cement in different proportions which gives strength values comparable to that of portland but is superior in resistance to chemical corrosion. In brief the aim of this work was to study.

(1) Effect of grinding time on apparent density, tap density, surface area and strength\* of Ashmoh cement.

(2) Effect of shape and composition of the grinding media on apparent density, tap density surface area and strength of Ashmoh cement.

(3) Effect of charge weight in the ball mill on strength.

(4) Effect of blending and interground additive on strength.

\* Strength test was done on 1:3 cement sand mortar discs.

- (5) Effect of water/cement ratio on strength.
- (6) Corrosion resistance of portland rice husk ash cement to various inorganic and organic acids.



## CHAPTER 4

### MATERIALS AND METHODS

#### 4.1 Materials:

Materials used in these experiments were rice husk ash, lime, sand, portland cement and organic and inorganic acids.

##### 4.1.1 Rice Husk Ash:

Rice husk ash was procured from a rice mill in Uttaripura, a village 60 km. from Kanpur on the G.T. road. The samples of ash available had different colours, black, grey and white. The grey ash, which is available in largest amount was used. Its carbon content was found to be 1.8% . Its bulk density was .213 gm/c.c. and tap density was .276 gm/c.c.

##### 4.1.2 Hydrated Lime:

Hydrated lime was obtained by sprinkling water (50% of weight of lime) on bed of quick lime lumps bought from the local market of Kanpur. To ensure complete hydration the bed was kept covered with moist gunny bags for two hours. After that it was dried up in oven and stored in air tight polythene bags.

### 4.1.3 Sand:

The sand used was locally available Kalpi sand. It was sieved (-16 mesh) and washed with water to get rid of all the clay content. Its bulk density was 1.4 gm/c.c. and tap density 1.61 gm/c.c.

## 4.2 Methods:

### 4.2.1 Grinding:

#### 4.2.2 Ball Mill:

For grinding a ball mill was used with following parameters

Height of the ball mill = 21 cm.

Internal dia of the ball mill = 28.5 cm.

Outer dia of the ball mill = 31.81cm.

Volume of the ball mill = 13.34l (directly measured)

and 13.6l (calculated)

The optimal range of RPM for the ball mill was fixed according to following formula:

$$\text{RPM}_{\text{minimum}} = \frac{76.63}{\sqrt{D}} \times \frac{3}{4}$$

$$\text{RPM}_{\text{maximum}} = \frac{76.63}{\sqrt{D}} \times \frac{4}{5}$$

where

D - Internal dia of ball mill (in ft.)

In the present work all the grinding was done at 60 RPM which was within the optimum range.

#### 4.2.3 Grinding Media:

Amount of grinding media was fixed so as to fill 40% of the ball mill volume. Different grinding media were used and they were

- (i) Small cylinders - Height = 1.250 cm.  
dia = 1.256 cm. weight = 12.8 kg.
- (ii) Large cylinders - Height = 2.072 cm.  
dia = 2.065 cm weight = 14.00 kg.
- (iii) Small spheres - size range = 1.10-1.14 cm dia  
Weight = 11.08 kg
- (iv) Large spheres - size range = 1.924-1.962 cm dia  
weight = 11.35 kg.

Rice husk ash and hydrated lime were charged into ball mill in determined amounts along with the specific grinding media. The mill was run for required amount of time at a fixed speed.

#### 4.2.4 Discs and Cubes Samples:

Rings of 7 cm. internal dia and 3.5 cm height were used as moulds to make disc of compacted mix samples. These discs were used to determine tensile strength by cylinder splitting test.

Brass split moulds (face area  $50 \text{ cm}^2$ ) were used to make cube samples.

(a) Mixing Procedure:

Ashmoh cement and sand were mixed in batches to get a uniform mixture. Water was added to it in measured quantity and mixed thoroughly to get a uniform mix. The mix was covered with a moist cloth to maintain the same water content while making discs or cubes. The mix was hand moulded.

(b) Hardening:

The sample were kept under a moist cloth for 48 hours to complete initial hardening.

(c) Curing:

After initial hardening samples were either water cured or steam cured for further strength development.

In water curing samples were immersed in water for 28 days at room temperature.

In steam curing samples were kept in saturated steam at 1 atm for 10 hours to reach nearly the same 'maturity' stage as accomplished by 28 days water curing as determined by Jain.

### 4.3 Testing

#### 4.3.1 Apparent density, Tap density and True density:

Apparent density was measured by loosely filling a beaker of known volume. Excess material on the top was levelled off and amount was found out. From the known mass and volume apparent density was computed.

Tap density was measured by taking appropriate amount of material in a one litre measuring cylinder. Sample was tapped on a vibratory table until there was no further compaction in the bed. Volume was directly read on the measuring cylinder.

True density was determined by 'specific gravity bottle' method. Acetone was used for Ashmoh cement.

#### 4.3.2 Surface Area:

Blain apparatus was used to determine specific surface area of Ashmoh cement. The formula used is

$$S = S_s \frac{e_s(1 - e_s)}{(1 - e)} \frac{\sqrt[3]{e_s^3}}{e_s^3} \frac{\sqrt[3]{n_s}}{\sqrt[3]{n}} \frac{\sqrt{T}}{\sqrt{T_s}}$$

where,

S : specific surface area in sq.cm/g of test sample

$S_s$  : specific surface area in sq.cm/g of the standard sample used in calibration of the apparatus.

Here ordinary portland cement was used.

$T$  : measured time interval in seconds of manometer drop for test sample.

$T_s$  : measured time interval in seconds of manometer drop for standard sample used in calibration of the apparatus.

$\eta$  : viscosity of air in poise at the temperature of test of the test sample.

$\eta_s$  : viscosity of air in poise at the temperature of test of the standard sample (portland cement) used in calibration of the apparatus.

$e$  : porosity of prepared bed of test sample.

$e_s$  : porosity of prepared bed of the standard sample (portland cement)

$G$  : specific gravity of test sample (for Ashmoh)

$G_s$  : specific gravity of standard sample used in calibration of the apparatus (assumed to be 3.15 for portland cement).

If the measurements are taken at the same temperature and the same bed porosity is used ( $0.5 \pm .005$ ) we have the simplified eqn.

$$S = S_s \frac{\eta_s}{\eta} \frac{\sqrt{T}}{\sqrt{T_s}}$$

#### 4.3.3 Splitting Test:

Cylindrical discs of Ashmoh, sand mix are placed between two plates of testing machine such that their axis remains horizontal. Compressive load is increased till the failure takes place by splitting along vertical diameter.

The strength determined in the splitting test is believed to be closer to the true tensile strength of the concrete. The splitting occurs due to tensile strength equal to

$$T.S. = \frac{2P}{\pi HD}$$

where

P = compressive load on the cylinder

H = height of cylinder

D = diameter of cylinder

Precaution was taken to maintain a lowest possible speed of head throughout the testing.

#### 4.3.4 Cube Compressive Strength:

Cubes were tested in Universal Testing Machine. The load at which failure occurs divided by the face-area of the cube gives the cube compressive strength of mortar or concrete.

### Setting Time:

The setting time tests require the preliminary determination of the amount of mixing water required to produce a cement of standard consistency which was determined in the standard mould under the Vicat plunger. The usual range of values for Ashmoh was found to be between 45 to 50 water content.

The setting times were measured using Vicat apparatus with different penetration attachments as per the standard procedure.

### 4.4 Corrosion Resistance Test:

28 days water cured pellets were immersed in various solutions for corrosion test. The solutions used for this test were

- (i) 5%  $\text{H}_2\text{SO}_4$  solution.
- (ii) 5%  $\text{HCl}$  solution.
- (iii) 5% Acetic acid solution
- (iv) 5%  $\text{MgSO}_4$  solution
- (v) 5%  $\text{Na}_2\text{SO}_4$  solution
- (vi) 5% Ammonium sulphate solution

Solutions were prepared in distilled water.



At regular intervals, change in height, change in diameter and weight loss were recorded. Strength test were done on the samples after 60 days of immersion in the solution.

## CHAPTER 5

### EXPERIMENTAL RESULTS AND DISCUSSION

Grinding operations were carried out using grinding media of varying shapes and compositions. The aim was to determine optional grinding, parameters, that would lead to productivity maximisation, keeping capital investment and working time at minimum, when scaled up for a commercial venture.

In previous work effects of various additives have been studied, with portland cement being found the most suitable. In this different levels of portland cement replacement were tried so as to determine the optimum composition of ASHMOH.

It was expected that RHA-PORT cement, due to its lower line content, would have much better chemical resistance to corrosion when compared with normal portland cement. Experiments were carried out to evolve a new quality of RHA-PORT cement that would have better resistance to chemical attack. Later on in this work it is shown that RHA-PORT cement does have a higher resistance to corrosion as expected.

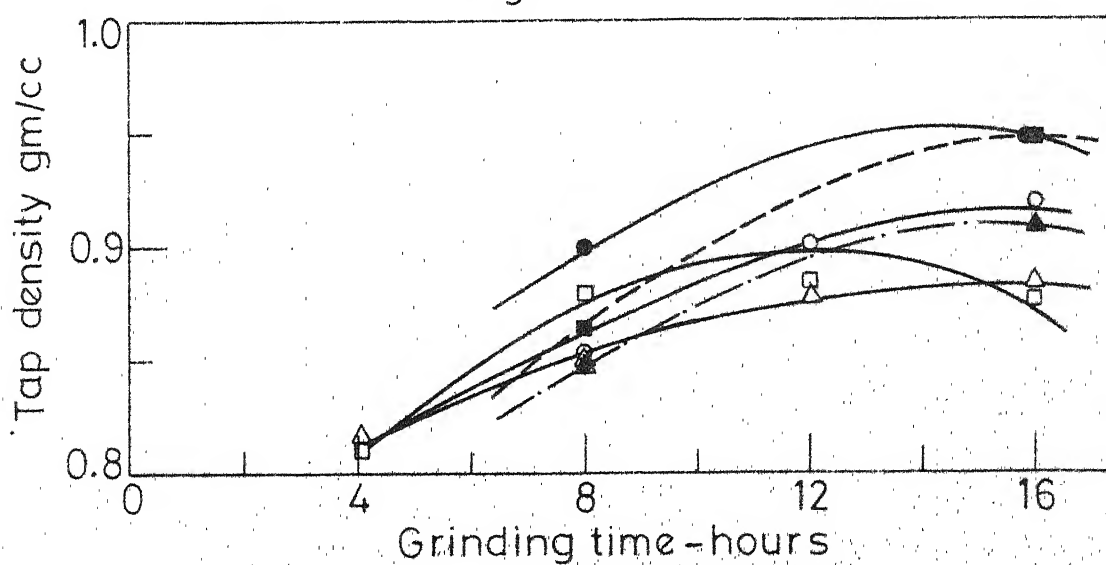
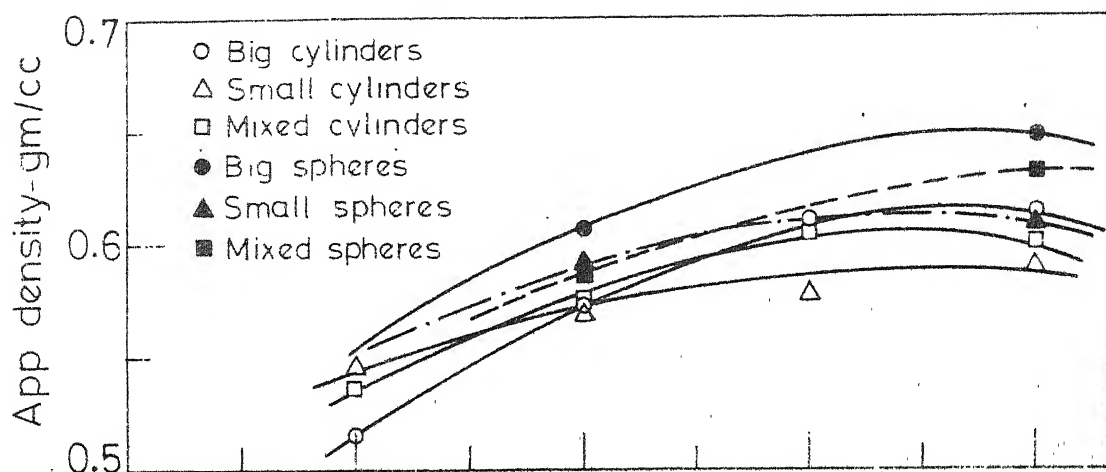
Ultimate tensile strength is used throughout this work for the purpose of comparison.

5.1 Effect of Grinding Time (G.T.) and shape of Grinding Media on Apparent Density (A.D.), Tap Density (T.D.), Specific Surface Area (S.S.A.) Tensile Strength (T.S.) and Bulk Density (B.D.)-

A 1:3 lime to RHA ratio gives maximum tensile strength in ASHMOH<sup>(14)</sup>. This strength is comparable to that of portland cement and hence all grinding operations were carried out with a standard 1:3 composition. Grinding time was varied in arithmetic progression of 4,8,12, 16 hours. Grinding media used were either spherical or cylindrical and of large and small and a mixture of large and small sizes.

Samples of ASHMOH were obtained for varying grinding media as mentioned above. These samples were then tested so as to determine A.D., T.D., S.S.A., for the purpose of primary evaluations. Pellets were made from different samples and these were tested to determine T.S. and B.D. The T.S. data was used to confirm the results of the primary evaluations obtained from the data of A.D., T.D., and S.S.A.

A.D. and T.D. initially increase with an increase in grinding time (Figs. 5.1.1 and 5.1.2). However, further increase in grinding time results in a decrease in the rate of increase of A.D. and T.D. This



Effect of grinding time on A.D. and T.D. of Ashmoh.

tendency of the curve to flatten out is consistent for different media shapes and sizes.

These results show that A.D. and T.D. are functions of particle size distribution at various stages of the grinding operation. Initially the concentration of coarse particles is higher-resulting in poorer packing of particles and hence lower A.D. and T.D. As grinding time is increased, the particle size distribution becomes wider with both finer and coarser particles being present. This results in a better packing of particles and consequently higher A.D. and T.D. Further increase in grinding time results in narrowing of the particle size distribution with the formation of porous agglomerates and hence A.D. and T.D. tend to level off-and even decrease.

The effect of grinding time on S.S.A. for different grinding media shapes and sizes results in a family of curves (fig. 5.1.3) similar to the curves obtained for A.D. and T.D. The data obtained conforms to the fact that S.S.A. increases as a function of particle size reduction. Thus, as grinding time is increased, reduction in particle size results in a higher value of S.S.A. However, increasing grinding time beyond a certain level leads to agglomeration of very fine particles which now form particles of enhanced size. This

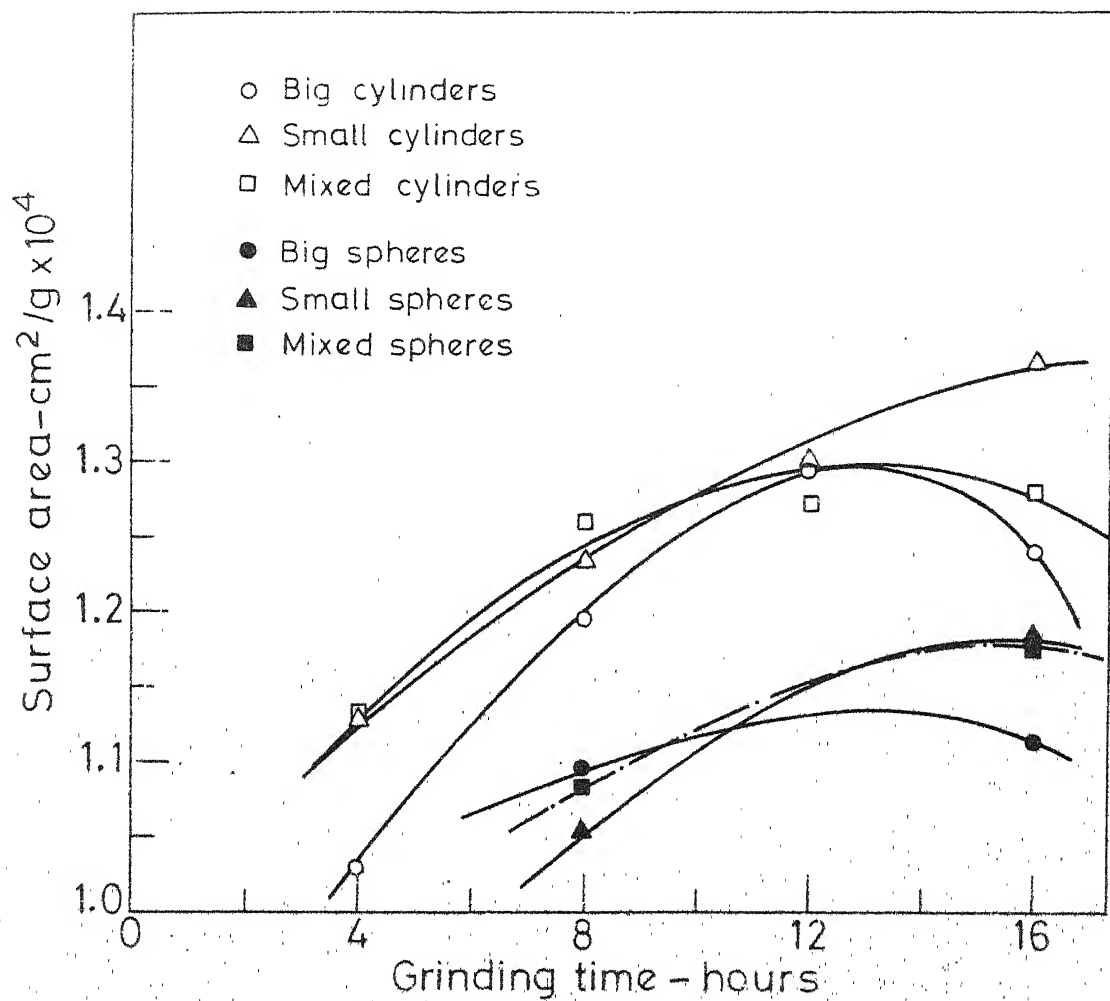


Fig. 5.1.3 - Effect of grinding time on S.S.A. of Ashmoh.

results in a drop in S.S.A. values due to higher grinding times. Higher S.S.A. results in an increase in the rate of formation of hydration products which renders strength to the cement. This may also lead to shorter initial and final setting time.

Pellets formed using the different samples of ASHMOH were tested to obtain data on T.S. and B.D. The T.S. curves (Fig.5.1.4) show that the T.S. increases with increasing grinding time until a certain point and the values either remain constant or start decreasing. This trend qualitatively conforms to the data on A.D., T.D., and S.S.A. which was obtained earlier.

B.D. curves (Fig. 5.1.5) exhibit very gradual increase with grinding time upto a certain point. The curves then flatten out or drop off. However, the data obtained for B.D. shows that the variation with grinding time is not very pronounced. This conforms to the T.S. data in that there is sufficient correlation between their values. Higher values of T.S. correspond to higher values of B.D.

Study of the various curves obtained (i.e. Figs. 5.1.1 to 5.1.5) shows the effect of the shape, size, and composition of grinding media and the important role these factors play in determining the quality of the cement.

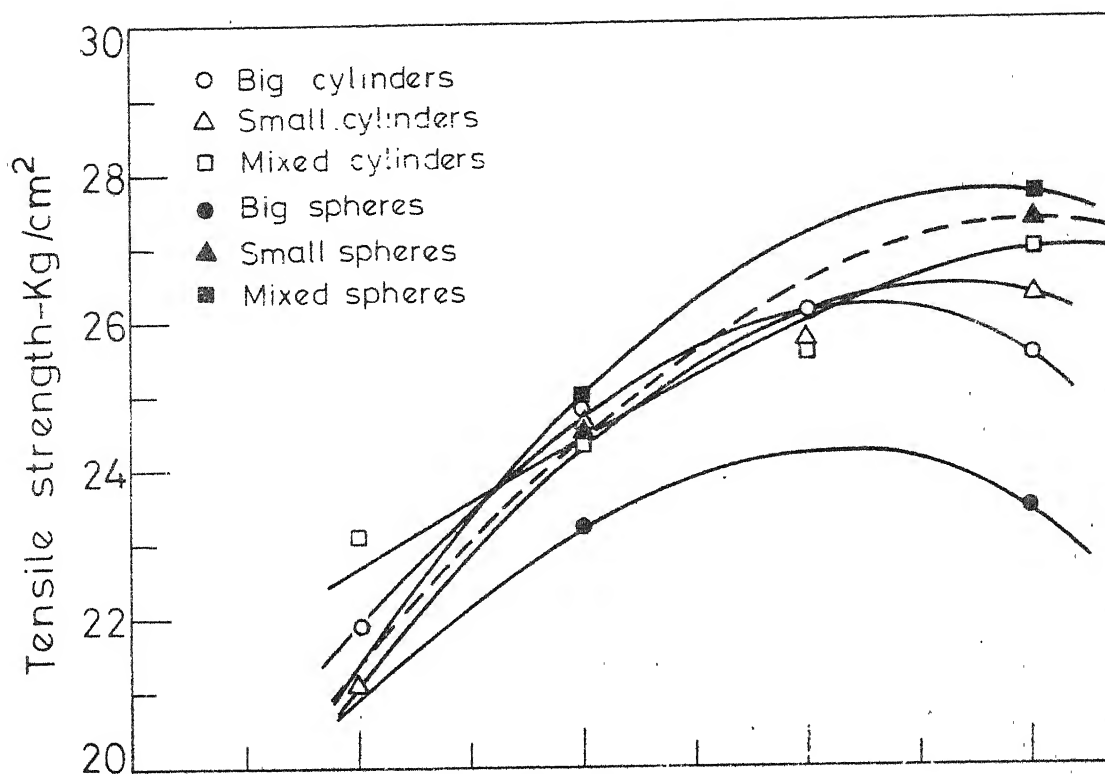


Fig. 5.1.4

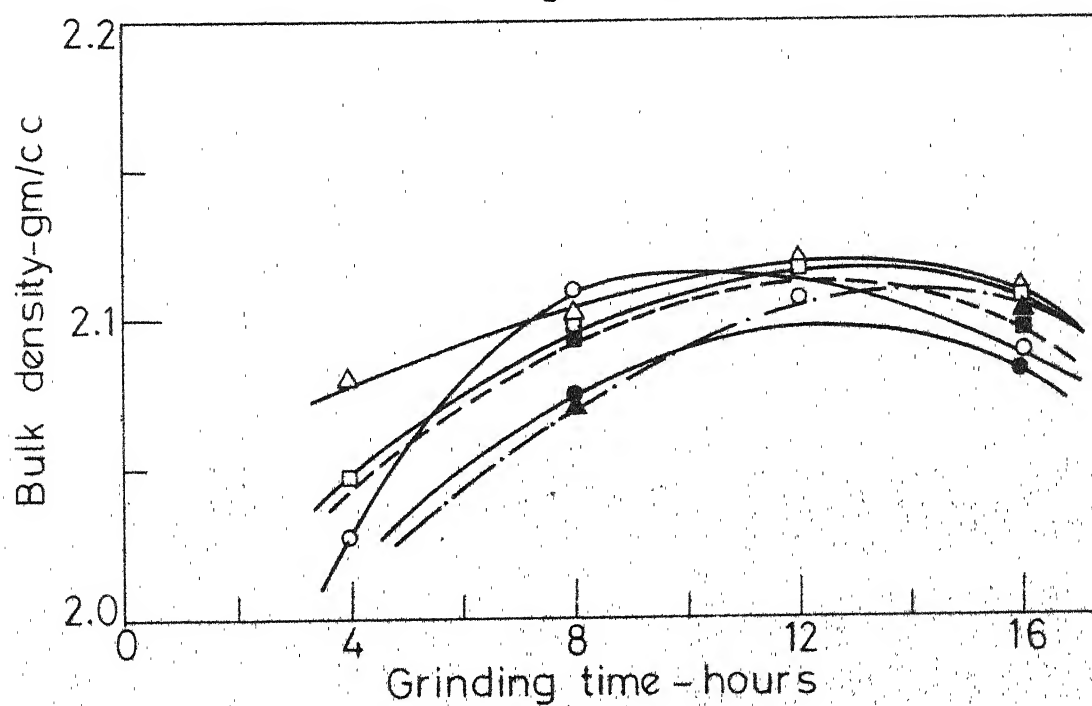


Fig. 5.1.5

Effect of grinding time on T.S. and B.D. of  
Ashmoh - sand mortar



This effect is best illustrated by the results obtained for T.S. tests. Selection of a grinding time of 8 hours with mixed balls or large cylinders results in the best combination of results. Further experiments were done with 8hr/large cylinders combination.

#### CONCLUSION:

- i) Grinding for large periods of time is not necessary as this does not lead to the best quality cement. The tendency of the curves to level off shows that the optimum grinding time is about 8 hrs. From the commercial point of view this time selection is very desirable.
- ii) Taking 8 hours as the optimum time period for grinding, the use of large sized cylindrical grinding media achieves near best results on T.S. Indeed, the T.S. achieved of  $24.5 \text{ Kg/Cm}^2$  is comparable to that of Portland Cement.

#### 5.2 EFFECT OF CHARGE WEIGHT IN THE BALL MILL ON STRENGTH:

In this test it is sought to optimise the charge weight that will lead to the best results of T.S. etc. while maintaining the productivity of the process.

For this purpose, different specimens of ASHMOH were prepared using different charge weights. Each run

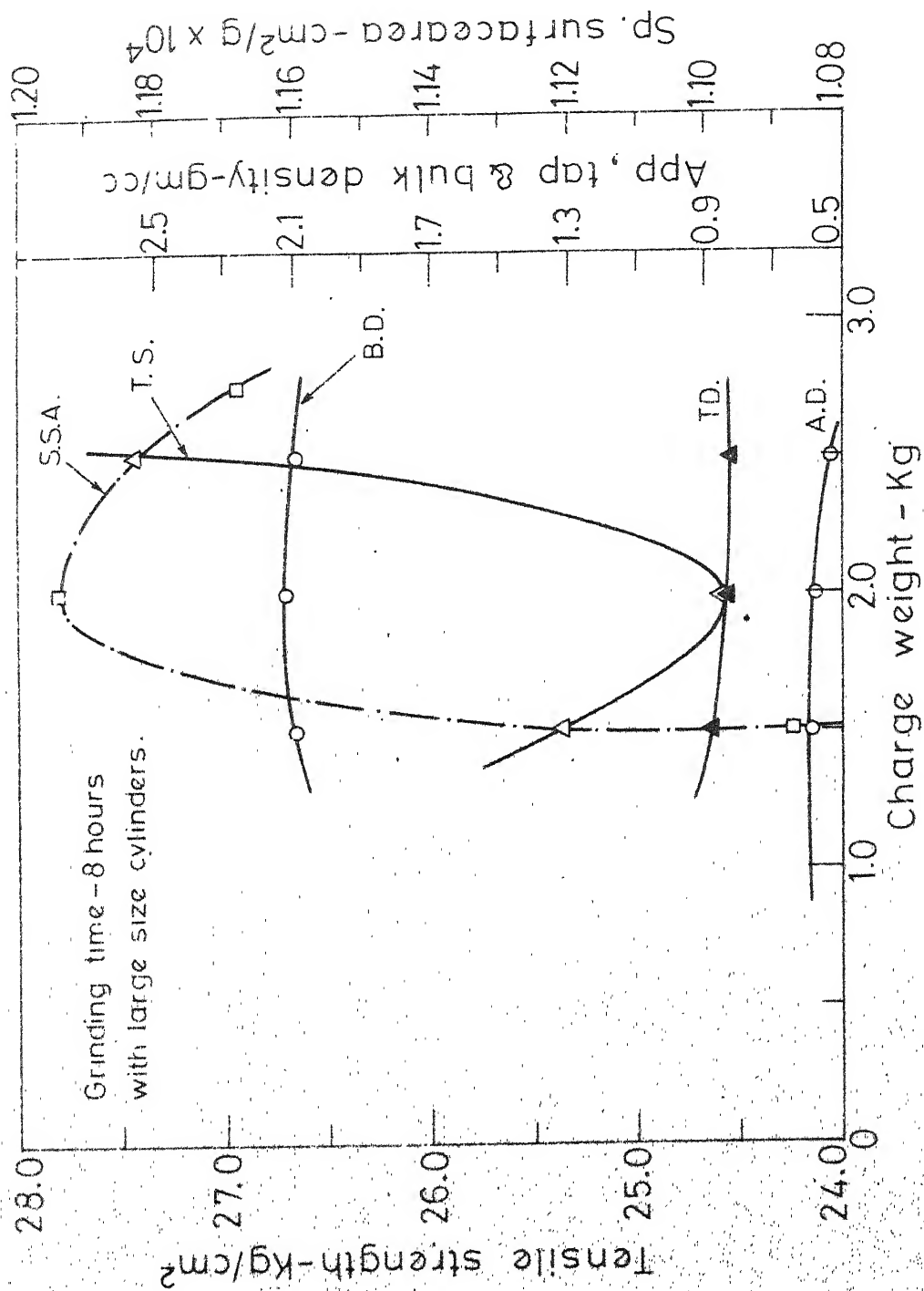


Fig 5.2 - Effect of charge weight on A.D, T.D, S.S.A., T.S. and B.D.

was for 8 hrs and large cylindrical grinding media were used (From 5.1). In the range tested, it was found that the curves of A.D., T.D., and B.D. are comparatively flat with nominal changes in strength (Fig. 5.2).

The maximum value of tensile strength was obtained when the charge weight was 2.5 Kg. which corresponded to 0.184 Kg. charge/litre mill capacity. All further experiments were done at this charge loading. T.S. results shown in Fig. 5.2 are comparable to those for Portland Cement.

However, it is not possible to explain the strange behaviour of the T.S. curve as against the S.S.A. curve.

### 5.3. EFFECT OF PORTLAND REPLACEMENT ON T.S.:

The effect of Portland cement as an additive was investigated with the aim of further increasing the strength of ASHMOH. For a quick comparasion T.S. Data was obtained by breaking steam cured samples that had been hand blended with varying percentages of portland replacement. To reinforce the results obtained, additional samples with Portland replacement and varying w/c ratio were water cured and the T.S. data obtained. The effect of Portland replacement was also studied by mixing and grinding it with RHA and lime in the grinding stage.

### 5.3.1 STEAM CURED SAMPLES (Blended)

Steam curing at 1 atm. pressure for 10 hours gives strength which is approximately equal to that of the samples which are water cured for 28 days (JAIN). Therefore steam curing is of considerable help in carrying out a quick comparative study. However, it does not provide an exact indication of strength values. In steam curing, the strength depends heavily upon the W/c ratio. (A lower w/c ratio in the sample results in better strength).

The data obtained in this work (Fig.5.3.1) shows. that the T.S. increases with an increase in the percentage of Portland replacement. Bulk density also increases with the increase in percentage of portland replacement. This is in conformity with the results obtained from T.S. data.

### 5.3.2 WATER CURED SAMPLES (Blended)

From the data obtained from steam cured samples, it was concluded that an increase in the percentage of portland replacement results in increased T.S. of the cement. In this work, more rigourous experimentation was carried out using samples with varying w/c ratios that had been water cured for 30 days. The objective is to study the effect of portland replacement on T.S. for different w/c ratios.

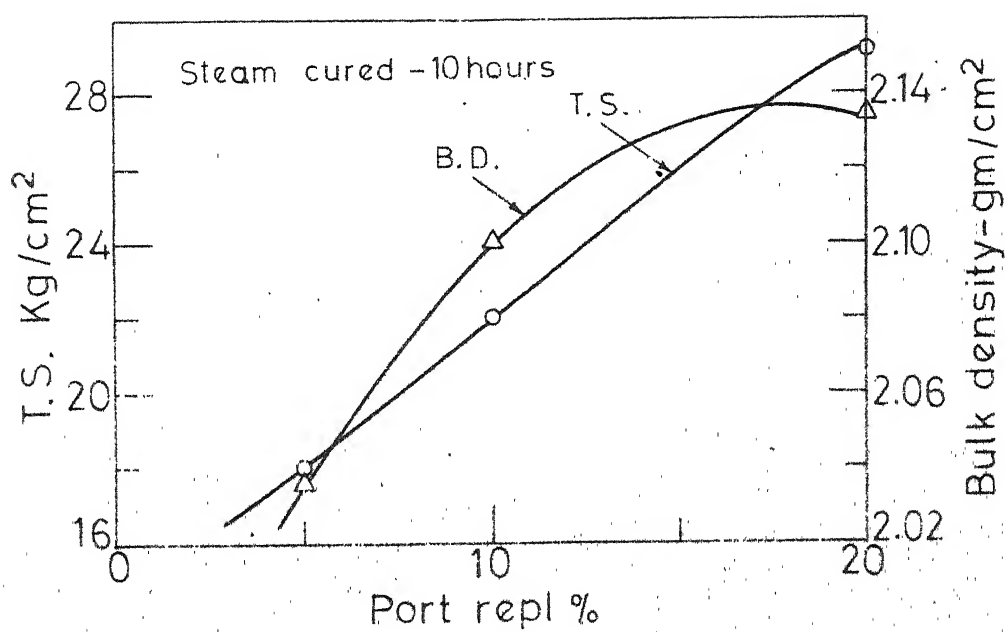


Fig. 5.3.1 - Effect of portland replacement on T.S. and B.D. (Blended)

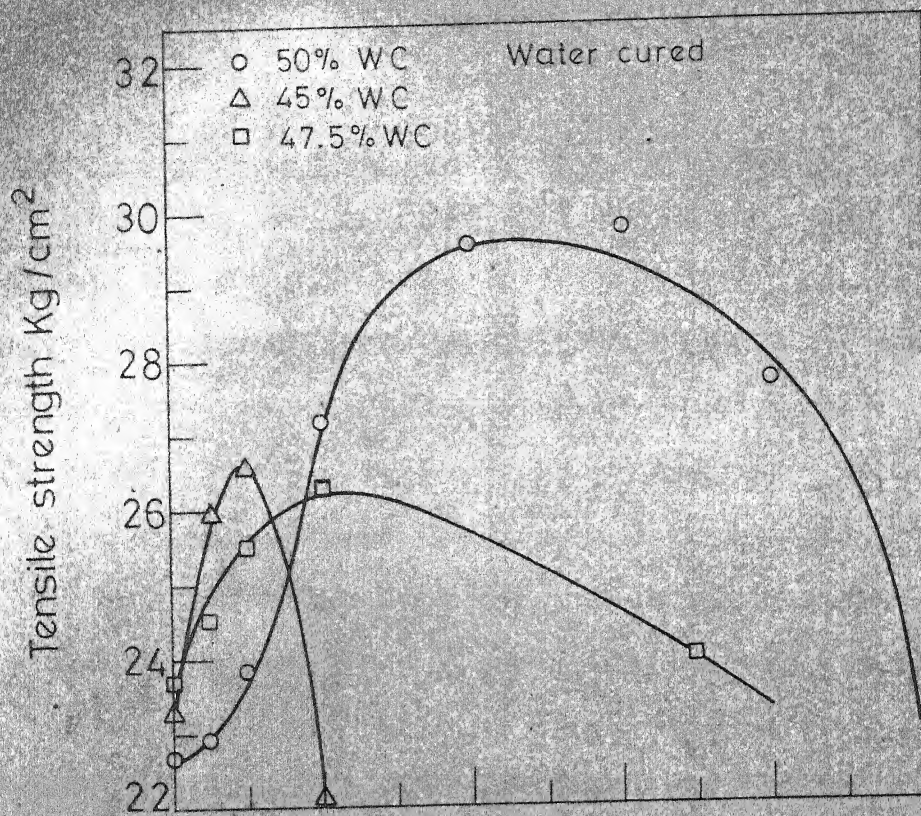


Fig. 5.3.2

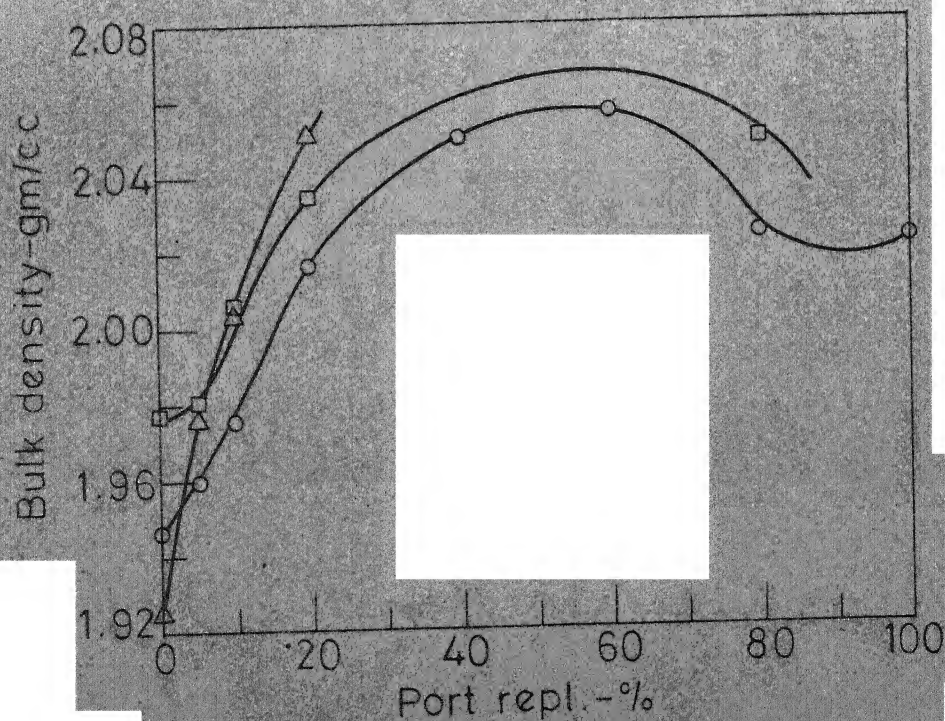


Fig 5.3.3

Effect of portland replacement with varying W / C ratio on T.S. and B.D.

Data was obtained (Fig. 5.3.2) for w/c ratios of 0.45, 0.475, and 0.50. For w/c ratio of 0.45, the T.S. kept increasing till 10 per cent portland replacement after which it starts decreasing whereas the B.D. keeps increasing. For w/c ratio of 0.475 the T.S. increases upto 20 per cent of portland replacement. B.D. data (Fig. 5.3.3) fairly conforms to the T.S. data. For w/c ratio of 0.50, the T.S. increases upto 50 per cent portland replacement, then decreases. Here too the B.D. data conforms to the data obtained for T.S. We note that the early portions of these curves match the results obtained from steam cured samples, although the values of T.S. obtained are lower.

### 5.3.3 EFFECT OF PORTLAND REPLACEMENT (INTERGROUND) ON T.S.

So far, the effect of portland replacement (blended) on T.S. was studied. It is assumed that intergrinding will result in a more homogenous mixture leading to an evenly compacted mass with higher strength values. In this case 5 per cent and 10 per cent of ASHMOH were replaced by portland cement and then ground in the ball mill. Samples were taken after 4, 6, and 8 hours.

Figure 5.3.4 illustrates the T.S. data as a function of grinding time. It is observed that T.S. increases as the grinding time is increased. Also, the strength values are higher for 10 per cent portland

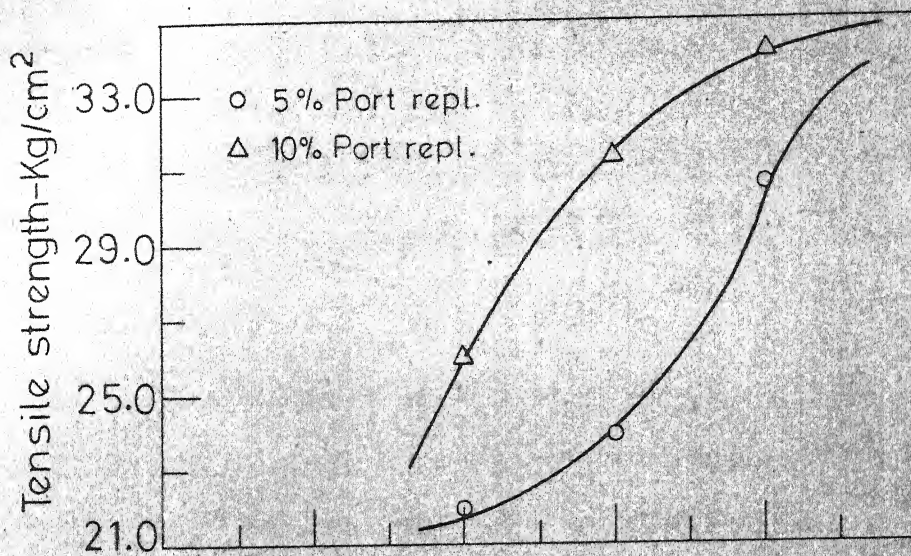


Fig. 5.3.4

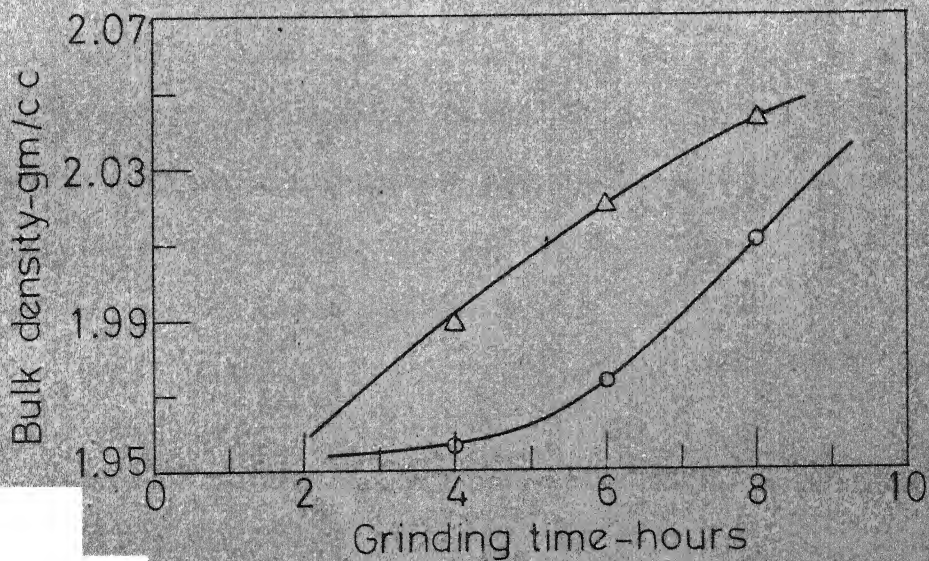


Fig. 5.3.5

Effect of grinding time on T.S. and B.D. (PORT interground).



replacement than for 5 per cent portland replacement. The B.D. data obtained conforms to the T.S. data. These results are as per the expectations (Fig. 5.3.5). The T.S. values obtained turned out to be higher than those when blending was done.

It is also observed that 5 per cent replacement with grinding time of 8 hrs gives the same strength value as 10 per cent replacement with a grinding time of 6 hours. The strength values obtained match that of portland cement.

When the process of manufacturing ASHMOH is scaled up for commercial plants, it would not be possible to maintain the stringent conditions of the Laboratory. Masons also do not rigidly adhere to the required specifications for its use in construction. Thus for safety purposes it would not be unreasonable to select 10 per cent portland replacement with 8 hrs grinding time as most suitable.

It is not possible to give a very definite explanation of the causes governing the effects of portland replacement on Ashmoh. Possible explanations for these causes are proposed as follows:

1. PARTICLE SIZE DISTRIBUTION: The T.S. of the cement mix is dependent upon the particle size distribution. LHA

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wider particle size distribution results in better packing of the particles and hence higher strength values. This may well be a major cause for the increase in T.S. as seen in the early portion of the curve.

2. COMPOSITION OF THE MIX: The strength properties of ASHMOH vary with the composition of its constituents. Commercially available Portland cement contains 66 percent of lime - all of which does not take part in the hydration reactions. The free lime thus available can affect the composition of ASHMOH. When the lime content is sufficiently high, all the amount is not expected to react with the ash and thus any excess lime may interfere with the formation of aggregate-cement bonds (BAJPAI). This deviation from the optimum amount of lime may well lead to a decrease in T.S. and the effect will become more noticeable as the Portland replacement percentage is increased.

3. THE W/C RATIO: The water requirement for setting of Portland cement is different from ASHMOH since the latter, with its higher S.S.A., is expected to need more water during hydration than Portland cement. The data has been obtained for different percentages of Portland replacement with fixed w/c ratio. Depending upon the W/c ratio, an optimum percentage of Portland replacement for which T.S. is maximum has been obtained in the data. As the percentage of Portland replacement is increased towards

the optimum, T.S. values will also increase. Also, beyond the optimum, the strength values may decrease. It has been noticed that at a certain W/c ratio, the mass is fully compacted. Increases in the water level results in a concrete mix of low consistency in which the coarse and fine aggregates tend to segregate - with the coarse aggregates setting towards the bottom during compaction and pouring. This segregation will result in a lowering of T.S.

Combination of the three possible causes cited above provide us with possible explanations for the data obtained. The initial rise in T.S. may be due to favourable particle size distribution and due to optimisation of w/c ratio. The effect of free lime on the aggregate-cement bonds is not sufficiently pronounced to offset the increase in T.S. attributed to the other two causes. Further increase in Portland replacement results in a levelling off of the effect due to particle size distribution. The effect due to quantity of free lime present becomes more pronounced and also the W/c ratio will lead to a decrease in T.S. These factors in combination lead to a decrease in T.S. beyond the optimum point.

For a commercial venture, portland replacement percentage is limited by the factor of cost and strength value requirements. The cost of Portland cement limits

the amount of additive. On the other hand, the strength value requirement limits the minimum amount of additive. Considering these factors and from results obtained, 10 per cent portland replacement seems justified.

#### 5.4 EFFECT OF PORTLAND REPLACEMENT ON T.S. OF ASHMOH WITH DIFFERENT COMPOSITIONS (INTERGROUND):

So far, all samples of ASHMOH used had 1:3 lime to RHA ratio. This is the optimum composition as determined by BAJPAI. In this work an attempt is made to study the effect of portland replacement on ASHMOH when a different composition ratio is taken. Here the attempt is made to decrease costs by selecting ASHMOH with 1:4 lime to RHA ratio and getting improved tensile strengths by Portland replacement (Interground) of 5 per cent and 10 per cent.

The data obtained shows that there is only a slight decrease in T.S. when compared to values with 1:3 composition (Fig. 5.4.1). Lime is a perishable item and is likely to deteriorate when it is stored in large quantities for scaled up production. Suppose if lime deteriorates by 20 per cent, then 1:4 composition will probably not allow any safety margin. Hence 1:3 composition is recommended as being more suitable.

Ashmoh with 10% portland cement replacement was tested for its setting times. Values obtained for initial and final setting times were 45 minutes and 5 hours respectively. Its compressive strength was found to be  $379 \text{ kg/cm}^2$  (Table 5.4).

Table 5.0  
Compressive Strength Values

Lime/Ash ratio	Portland Replacement	B.L <sub>1</sub>	B.L <sub>2</sub>	T.S <sub>Av</sub>
1:4	5%	15825	13100	289
1:4	10%	16700	19500	362
1:3	5%	15250	16250	315
1:3	10%	17250	20650	379

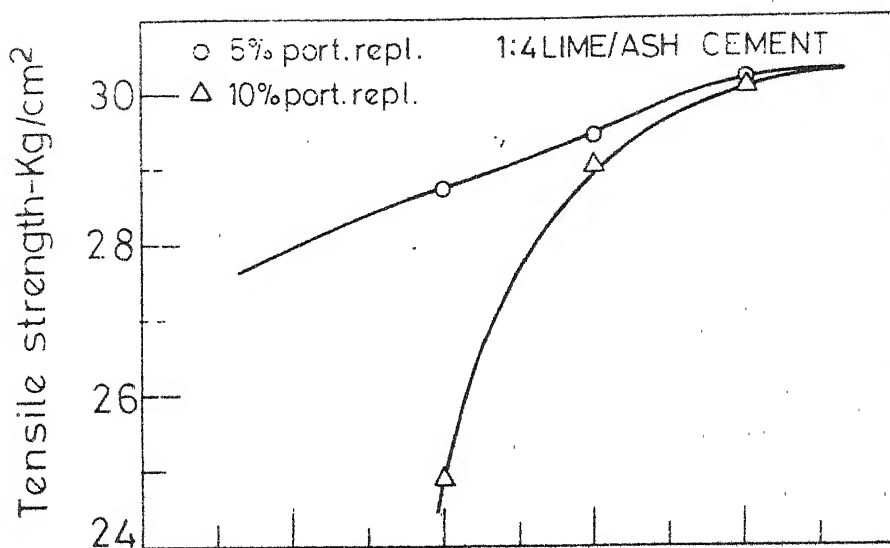


Fig. 5.4.1

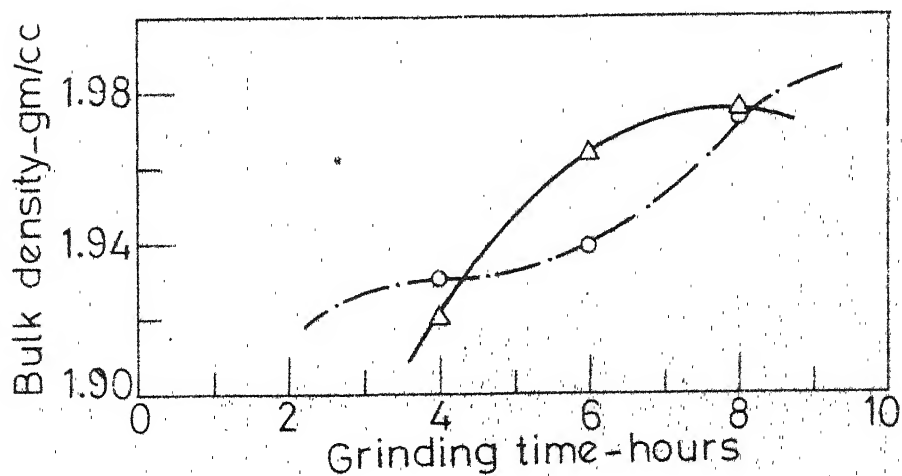


Fig. 5.4.2

Effect of grinding time on 1:4 on Lime/Ash ratio cement (PORT Interground).

### 5.5 RHA - PORTLAND CEMENT:

The aim of this part of the work is to develop a new quality of low cost cement cement with a high degree of chemical resistance to corrosion while maintaining high strength values.

When ground RHA is added to Portland cement it develops pozzolonic activity and provided sufficient time is allowed for setting, the cement has good chemical resistance. The RHA consumes the free lime in Portland cement, hence increasing its chemical resistance.

Fig. 5.6 shows an increase in T.S. of Portland cement when RHA (ground for 8 hrs) is added; and then, after an optimum point, the T.S. starts decreasing. This decrease is due to the insufficient amount of free lime that is required for reacting with the RHA.

An optimum composition for obtaining the best combination of chemical resistance and T.S. is an 1:1 ratio of Portland cement and RHA. Using previous results we know that optimum w/c ratio of 0.5 will lead to total optimisation in this case. That the strength is maxm. at 1:1 composition is not surprising since 100 parts of Portland cement generate 20 parts free lime after taking care of its hydration products and this amount of free



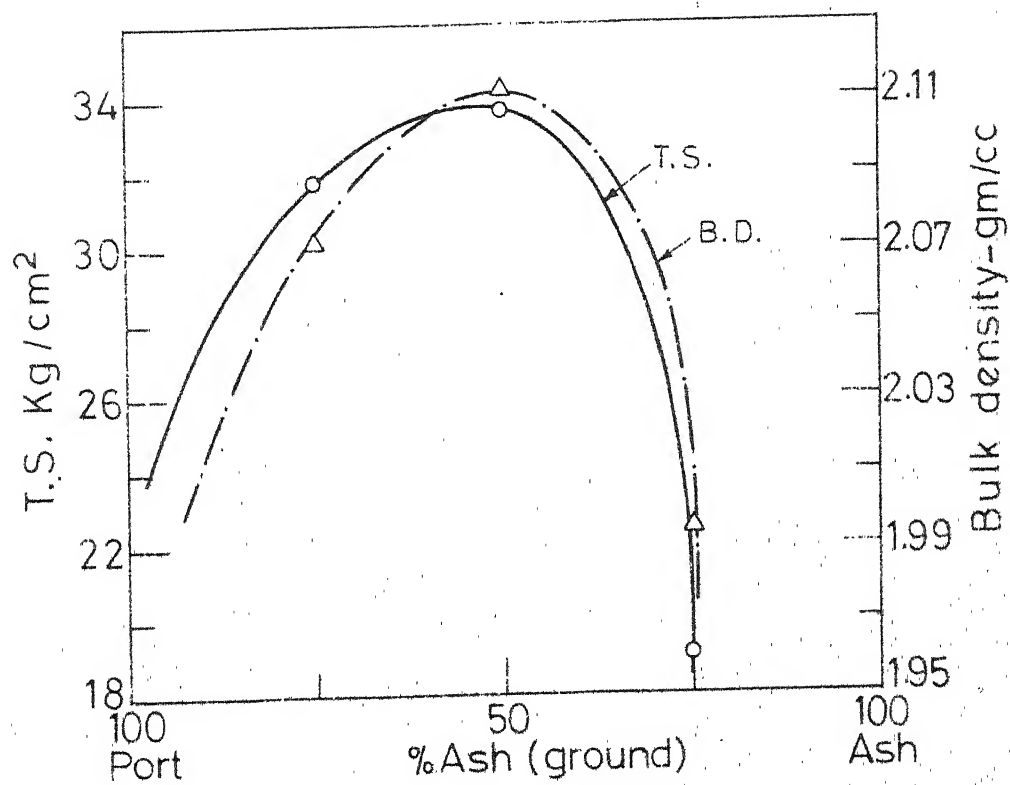


Fig. 5.5 - Effect of ground Ash replacement in portland on T.S. and B.D.

lime forms approximately 1:3 ratio with RHA. In other words this 1:1 RHA-PORT composition generates an ASHMOH composition in situ where the lime constituent is supplied by the Portland cement.

### 5.7 CHEMICAL ATTACK ON RHA-PORT CEMENT (1:1 RHA-PORT RATIO):

The effect of different chemical solutions on sand mortars made with RHA-PORT cement was studied by comparing it with the effect on portland cement. The percentage expansion or contraction or the percentage weight gain or loss were determined over a number of days for both RHA-PORT and portland cement (Figs. 5.7.1-5.7.5) Mortar discs were water-cured for a 28 days period and then immersed in turbulence-free solutions of different acids and sulphates. The tensile strength of pellets was measured after 60 days immersion in chemical solution.

#### RESULTS:

##### (a) HCl and Acetic acid:

The plot (Fig. 5.3.1 and 5.3.2.) for percentage contraction and percentage weight loss against number of days, for RHA-PORT immersed in HCl and Acetic acid, first increases and then tend to flatten out. A comparison with the effect on portland shows that percentage weight

loss is lower in the case of RHA - PORT cement. Besides this, Table 5.7 shows that RHA-PORT cement maintains higher strength properties than portland cement does.

(b) H<sub>2</sub>SO<sub>4</sub>:

Observation showed that both RHA-PORT and portland cement started disintegrating after 2 days of immersion in H<sub>2</sub>SO<sub>4</sub>. Disintegration was observed to be more for portland cement and less for RHA-PORT cement.

(c) Ammonium Sulphate:

Pellets of RHA - PORT cement first contracted and then started expanding whereas pellets of portland cement expanded continuously. RHA - PORT cement pellets initially registered gain in weight and subsequently started to loose weight. Portland cement pellets registered continuous gain in weight.

On drying after 60 days of immersion in sulphate solution cracks appeared in the portland cement pellets whereas no such cracking was observed in the RHA - PORT pellets.

(d) MgSO<sub>4</sub> solution:

RHA-PORT pellets initially contracted and then started to expand. Portland cement pellets also contracted initially but subsequently there was no

change with further passage of time.

Weight of the pellets first decreases and then increases gradually. Table 5.7 shows that after 60 days immersion the tensile strength of RHA-PORT is much higher than that of portland cement (T.5.7).

(e) Na<sub>2</sub>SO<sub>4</sub>:

Both RHA-PORT cement pellets and portland cement pellets contracted initially and then there was no change. Pellets of both cements first gained and then started to loose weight.

Tensile strength of RHA-PORT was higher than that of portland cements after immersion (T.5.7).

### Discussion:

Chemical attack can cause mortar to corrode in two ways:

(1) Surface Corrosion - Chemical reaction takes place at the surface of the pellets. Reaction products are washed away.

(2) Bulk Corrosion - The chemical solution permeates into the mass of the pellet and reacts with the cement there. Some part of the reaction products accumulate, resulting in volume changes.

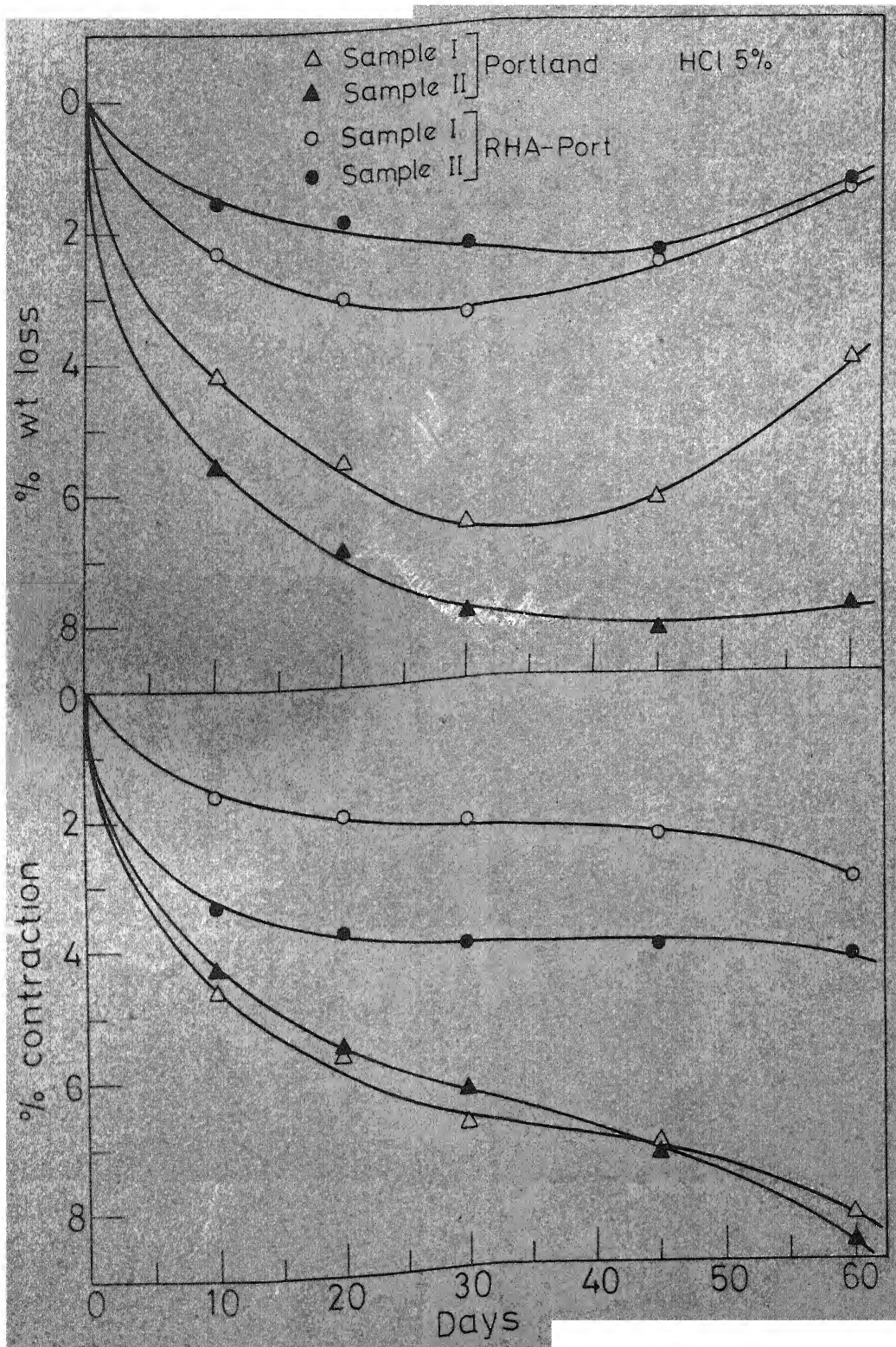


Fig. 5.6.1

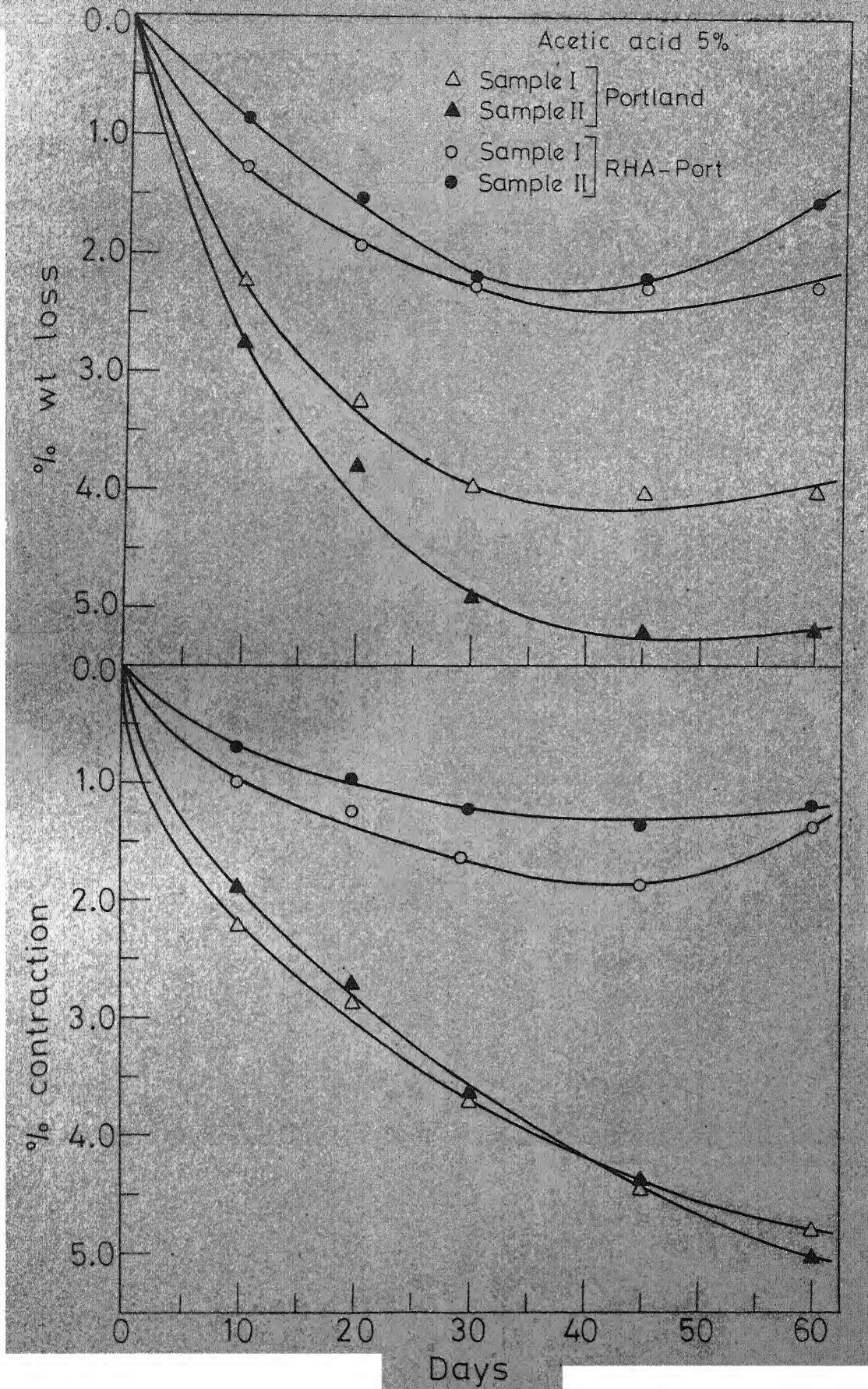


Fig. 5.6.2



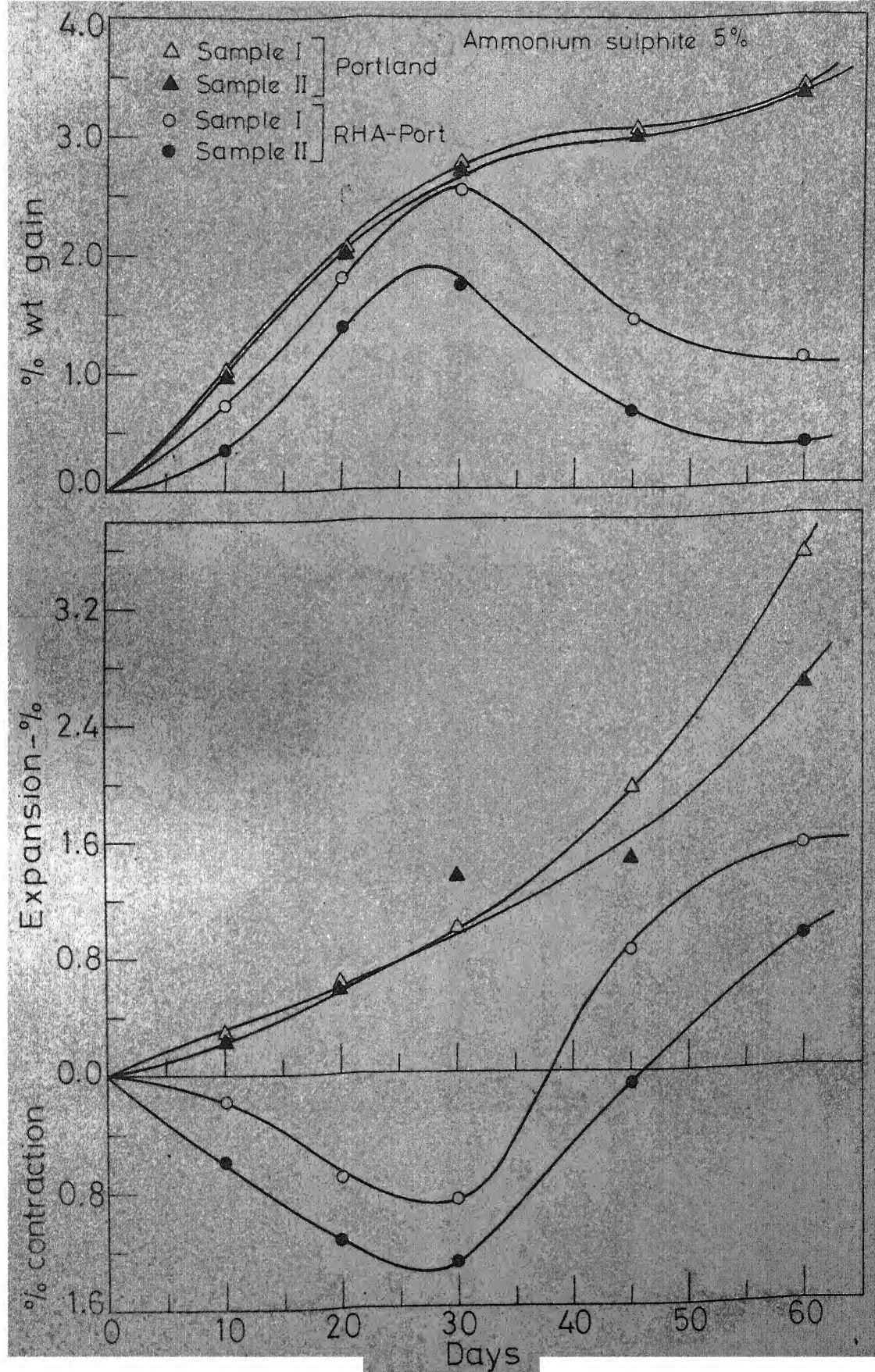


Fig 5.6.3

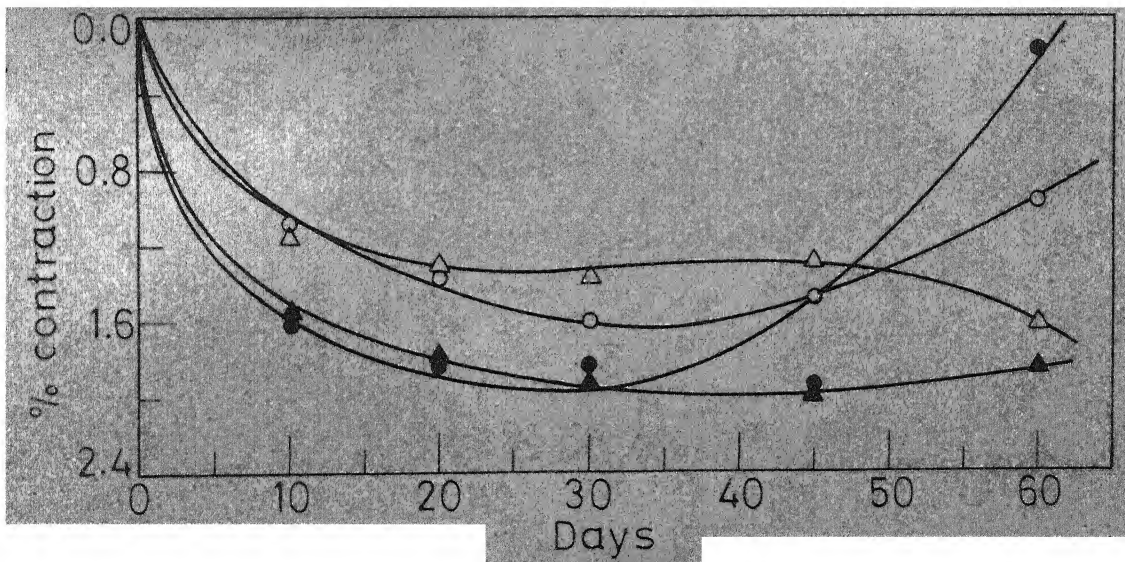
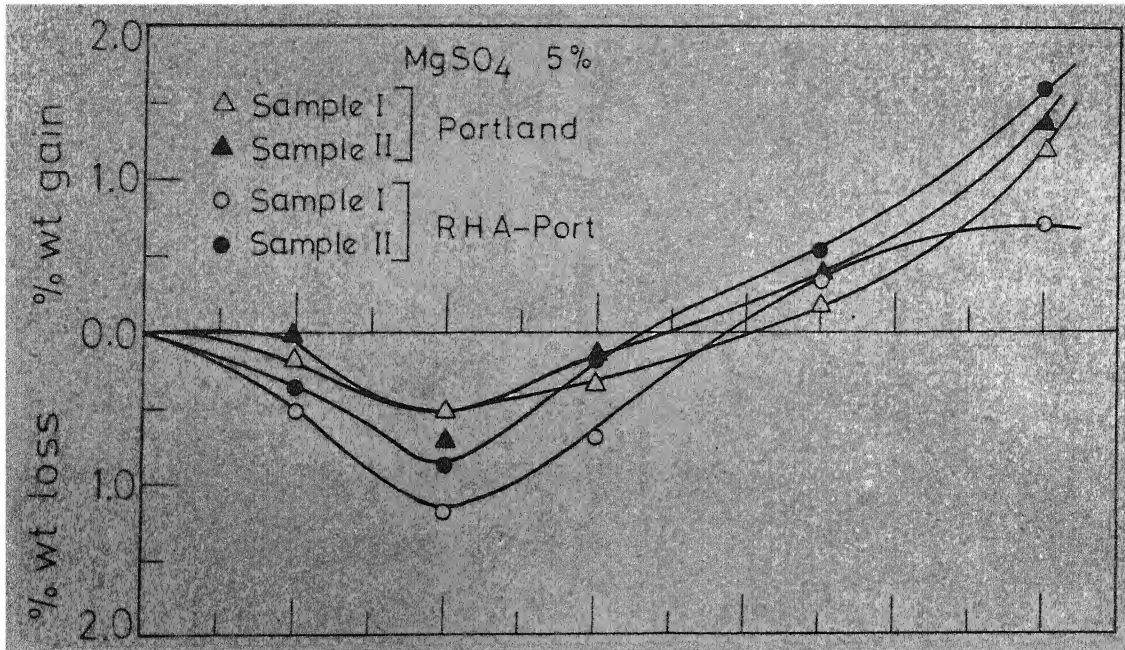


Fig. 5.6.4



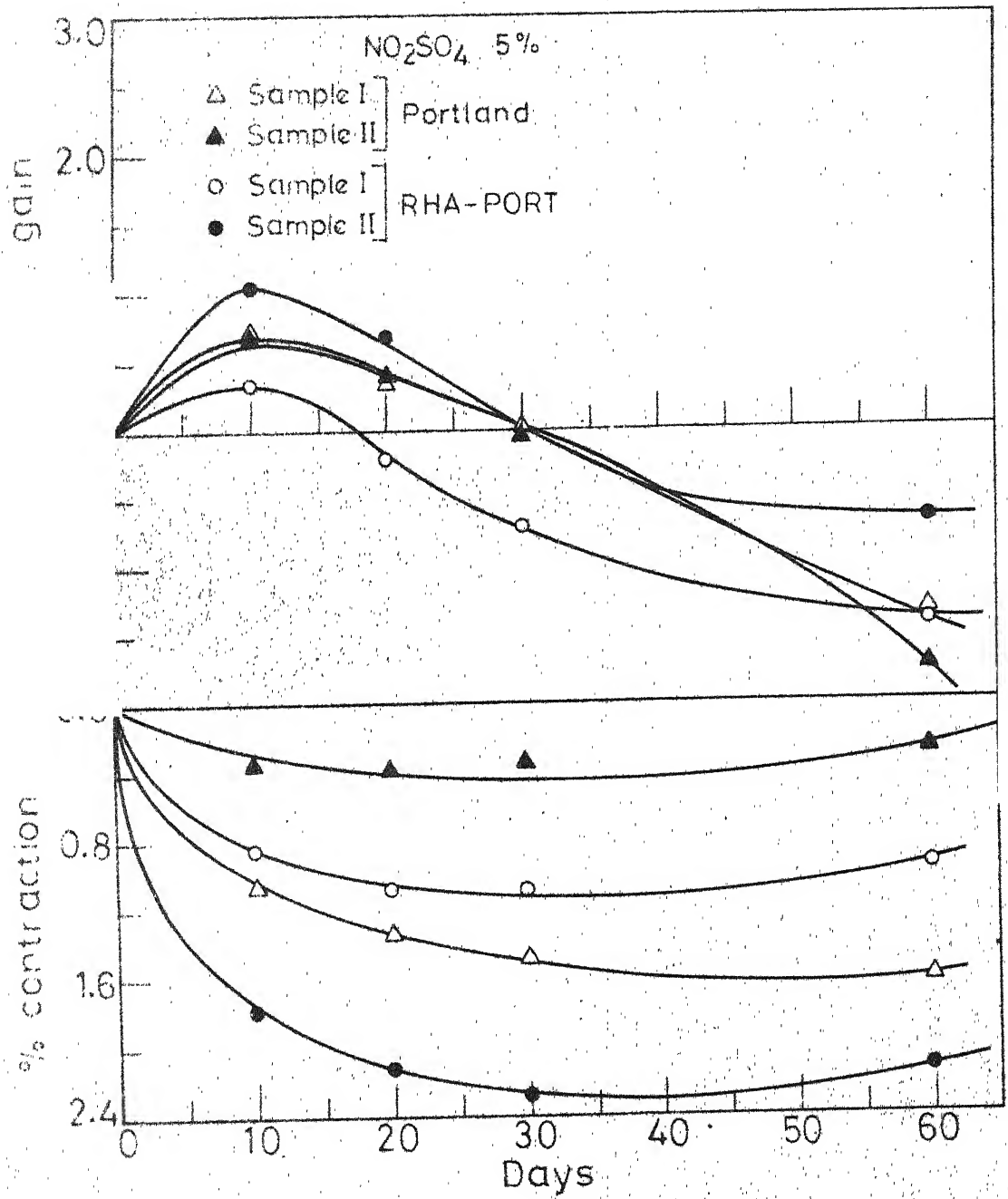


Fig 5.6.5

Table 5.7

Chemical Agent Sample		<u>RHA-PORT</u>		<u>PORTLAND</u>	
		<u>BL</u>	<u>TS</u>	<u>BL</u>	<u>TS</u>
HCl	I	1350	36.01	900	24.13
	II	4600	41.37	800	21.46
Acetic acid	I	1425	37.36	1050	27.57
	II	1550	40.08	840	22.32
MgSO <sub>4</sub>	I	1175	31.02	1250	32.5
	II	1200	31.34	1050	27.30
NaSO <sub>4</sub>	I	1125	29.78	1025	26.34
	II	1275	33.29	1070	27.57

## CHAPTER 6

### CONCLUSIONS

On the basis of the experimental work carried out and results obtained therefrom, the following conclusions can be drawn:

- (1) Optimum grinding is 8 hrs.
- (2) For 8 hrs. grinding time, most effective grinding media are large cylinders.
- (3) Optimum charge weight was found to be .184 Kg/litre mill capacity
- (4) Optimum portland replacement in ASHMOH is 10 per cent.
- (5) When ASHMOH is interground with 10 per cent portland replacement, lime/ash ratio found to give optimum results is 1:3.
- (6) 1:1 RHA/PORT ratio cement has substantially higher chemical resistance to corrosion than portland cement.

APPENDIX - 1CALCULATIONSA.1.1 BALL MILL

(a) Internal circumference of the ball mill = 90.4 cms

Height of the ball mill = 21.0 cms

$$\text{Volume of the mill} = \frac{(90.4)^2}{4 \times 3.14} \times \frac{21}{1000}$$

$$= 13.6 \text{ litres}$$

(b) Grinding media to be filled in is 40 of the mill volume.

Packing density for small cylinders

$$= \frac{12.8 \times 100}{13.6 \times 40} = 2.37 \text{ kg/litre}$$

Packing density for large cylinders

$$= \frac{14.00 \times 100}{13.6 \times 40} = 2.6 \text{ kg/litre}$$

Packing density for large spheres

$$= \frac{11.35 \times 100}{13.6 \times 40} = 2.1 \text{ kg/litre}$$

Packing density for small spheres

$$= \frac{11.084 \times 100}{13.6 \times 40} = 2.05 \text{ kg/litre}$$

(c) Speed of the mill

$$\text{RPM}_{(\text{max.})} = \frac{76.63}{\sqrt{D}} \times \frac{4}{5} \quad D - \text{Internal diameter in ft.}$$

$$= \frac{76.63}{\sqrt{90.4}} \times \sqrt{\pi} \times \frac{4}{5} \times \sqrt{2.54 \times 12} = 63$$

$$\begin{aligned} \text{RPM}_{(\text{min})} &= \frac{76.63}{\sqrt{D}} \times \frac{3}{4} \\ &= \frac{76.63}{\sqrt{90.4}} \times \sqrt{\pi} \times \frac{3}{4} \times \sqrt{2.54 \times 12} = 59 \end{aligned}$$

Speed chosen for the mill was 60 RPM

#### A.1.2 Characteristics of RHA and Sand

##### (a) Ash

(i) Apparent density Mass 65g

Bulk volume 305 c.c.

$$\text{Apparent density} = \frac{65}{305} = 213 \text{ kg/M}^3$$

(ii) Tap density Mass 138g

Tapped volume 500 c.c.

$$\text{Tap density} = \frac{138}{500} = 276 \text{ kg/M}^3$$

##### (b) Sand

(i) Apparent density Mass 425g

Bulk volume 305 c.c.

$$\text{Apparent density} = \frac{425}{305} = 1400 \text{ kg/m}^3$$

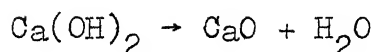
(ii) Tap density Mass 806g

Tapped volume 500 c.c.

$$\text{Tap density} = \frac{806}{500} = 1610 \text{ kg/cm}^2$$

### A.1.3 Composition of ASHMOH

$$\text{CaO/Ash ratio} = 1 : 3 \quad (a)$$



$$\text{Mol. weight of Ca(OH)}_2 = 74$$

$$\text{Mol. weight of CaO} = 56$$

$$\therefore x \text{ gm of CaO given } \frac{74}{56} x \text{ g of Ca(OH)}_2 \quad (b)$$

From (a) and (b)

$$3x + \frac{74}{56} x = 100 \quad (c)$$

$$x = \frac{100 \times 56}{242} = 23$$

Amount of Ash per 100 g mixture

$$= 69 \text{ g}$$

Amount of  $\text{Ca(OH)}_2$  per 100 g mixture

$$= 31 \text{ g.}$$

### A.1.4 Specific gravity measurements for ASHMOH

#### (a) With Xylene

Vol. of the specific gravity bottle = 50 c.c.

1. Mass wt. of empty sp. gr. bottle = 24.1069g

2. Mass wt. of sp. gr. bottle + Xylene = 66.771g

3. Mass wt. of sp. gr. bottle + Xylene (partially filled)  
+ ASHMOH cement = 21.227g

4. Mass wt. of sp. gr. bottle + xylene + cement = 21.227g

$$\text{sp. gr. of xylene} = \frac{42.6641}{50} = .8533$$

$$\begin{aligned}\text{Mass of cement} &= 54.2384 - 46.8808 \\ &= 7.3576\text{g}\end{aligned}$$

Mass of xylene displaced by cement

$$\begin{aligned}&= 42.664 - (71.227 - 24.1069 - 7.3576) \\ &= 74.1286 - 71.227 \\ &= 2.9016 \text{ g}\end{aligned}$$

$$\begin{aligned}\text{True density of ASHMOH} &= \frac{7.3576 \times .8533}{2.9016} \\ &= 2.1637 \text{ gm/cc.}\end{aligned}$$

(b) Bottle Toluene

1. Mass of the specific gravity bottle = 24.1061g
2. Mass of sp. gr. bottle + Toluene = 66.787g
3. Mass of sp. gr. bottle + cement = 36.6816
4. Mass of sp. gr. bottle + cement + Toluene = 74.3864

$$\text{sp. gravity of Toluene} = \frac{42.6709}{50} = .8538$$

$$\begin{aligned}\text{Mass of cement sample} &= 36.6814 - 24.1061 \\ &= 12.5753\text{g}\end{aligned}$$

vol. of (displaced) toluene

$$\begin{aligned}&= 50 - \frac{74.3864 - 36.6816}{.8538} \\ &= \frac{4.9852}{.8538} \text{ c.c.}\end{aligned}$$

$$\begin{aligned}\text{True density of cement} &= \frac{12.5755 \times .8538}{4.9852} \\ &= 2.1537 \text{ g/cc.}\end{aligned}$$

(ii)

1. Mass of sp. gr. bottle = 24.2837g
2. Mass of sp. gr. bottle + cement = 38.0382g
3. Mass of sp. gr. bottle + cement + toluene = 75.3575g

$$\begin{aligned}\text{Mass of cement} &= 38.0382 - 24.2837 \\ &= 13.7545\text{g}\end{aligned}$$

Volume of (displaced) toluene

$$\begin{aligned}&= 50 - \frac{75.375 - 38.0382}{.8538} \\ &= \frac{5.3707}{.8538}\end{aligned}$$

$$\begin{aligned}\text{True density of cement} &= \frac{13.7545 \times .8538}{5.3707} \\ &= 2.1766 \text{ gm/c.c.}\end{aligned}$$

Av. value of True density of

ASHMOH cement is = 2.165 g/c.c.



APPENDIX 2EXPERIMENTAL MEASUREMENTSAbbreviations

M	Mass in grams
H	Height in cm
D	Diameter in cm
V	Volume in $\text{cm}^3$
A.D	Apparent density gm/cc
T.D	Tap density gm/cc
S.S.A	Specific surface area $\text{cm}^2/\text{g}$
B.L.	Breaking load kg
T.S.	Tensile strength $\text{kg}/\text{cm}^2$
B.D.	Bulk density gm/cc
Av	Average
G.T	Grinding Time
RHA	Rice husk ash
T	time interval in seconds of manometer drop per test sample (Blaine Test)
W/C	Water to cement ratio
V	Volume charge, $\frac{v_t - v_o}{v_o} \times 100$
W	weight charge, $\frac{w_t - w_o}{w_o} \times 100$

## A.2.1 Charge 2 kg

Room Temperature =  $35^{\circ}$ - $36^{\circ}$ C

Mill speed = 60 RPM

Composition 1:3 Lime/RHA ratio

Grinding media required - to fill in 40 of the mill volume

 $T_s$  = 87 seconds (for portland cement)

G.T.	A.D.	T.D.	$T_1$	$T_2$	$T_3$	SSA (Av.)
<u>Large cylinders</u>						
4	.5147	.813	616	605	603	10272
8	.574	.853	891	893	903	11943
12	.610	.902	1065	1057	1056	12955
16	.613	.920	971	892	841	12403
<u>Small cylinders</u>						
4	.548	.814	789	796	796	11228
8	.568	.850	950	958	969	12325
12	.580	.879	1054	1050	1047	12901
16	.590	.885	1108	1112	1106	13673
<u>Mixed cylinders</u>						
4	.537	.810	795	799	806	11254
8	.574	.880	1004	1000	1003	12602
12	.606	.881	1020	1027	1028	12743
16	.60	.878	983	1031	1031	12781

Big spheres

8	.606	.90	757	763	750	10945
16	.646	.95	748*	778	787	11132

Small spheres

8	.590	.848	700	700	705	10544
16	.574	.910	900	881	880	11853

Mixed spheres

8	.587	.861	804	838	835	11264
16	.630	.950	796	802	805	11759

Charge weight 1.5 kg, large cylinders

8	.587	.896	748	736	751	10867
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Charge weight 2.5 kg, large cylinders

8	.531	.82	872	855	860	11687
---	------	-----	-----	-----	-----	-------

A.2.2 Charge weight = 2 kg

Composition of pellets - 1:3 cement/sand mortar

W/C .5

G.T.	H <sub>Av</sub>	D <sub>Av</sub>	M <sub>Av</sub>	BL <sub>1</sub>	BL <sub>2</sub>	BL <sub>3</sub>	T.S <sub>Av</sub>	B.D. <sub>Av</sub>
<u>Large cylinders</u>								
4	3.73	6.888	279.67	943	864	831	21.939	2.027
8	3.593	6.885	282.33	1015	995	855	24.573	2.111
12	3.633	6.866	283.67	788	1136	915	26.160	2.109
16	3.662	6.885	285.00	982	1066	972	25.426	2.090
<u>Small cylinders</u>								
4	3.571	6.877	276.00	832	700	900	21.024	2.080
8	3.607	6.888	282.33	968	<del>955</del>	<del>960</del>	24.136	2.101
12	3.610	6.865	283.33	1067	920	1020	25.791	2.120
16	3.611	6.865	282.33	960	1019	1096	26.324	2.112
<u>Mix cylinders</u>								
4	3.550	6.876	272.0	952	962	848	23.104	2.063
8	3.597	6.888	281.00	832	841	1060	23.408	2.096
12	3.639	6.869	285.33	1142	900	960	25.494	2.116
16	3.617	6.878	284.33	912	1076	1182	27.048	2.115
<u>Big spheres</u>								
8	3.613	6.888	279.33	1013	833	880	23.254	2.075
16	3.625	6.875	280.67	865	993	890	23.399	2.086

Small spheres

8	3.621	6.900	280.33	1082	873	934	24.535	2.070
16	3.700	6.881	289.67	1095	1136	1056	27.405	2.105

Charge weight = 1.5 kg. large cylinders

8	3.578	6.870	277.33	738*	998	962	25.381	2.091
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Charge weight 2.5 kg. large cylinders

8	3.579	6.887	278.33	1046	1120	1021	27.429	2.088
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A.2.3 Grinding time 8 hours

Grinding Media - Large cylinders

Charge weight - 2.5 kg

Port-H land	$H_{av}$	$D_{av}$	$M_{av}$	$BL_1$	$BL_2$	$BL_3$	$TS_{av}$	$B.D._{av}$
water cured		.5	water cured					
0	3.64	6.871	262.7	845	900	935	22.73	1.946
5	3.683	6.876	268.0	890	840	1000	22.88	1.960
10	3.593	6.872	263.3	925	920	920	23.85	1.976
20	3.637	6.879	272.7	1150	975	1075	27.23	2.017
40	3.651	6.872	277.3	1275	1220	1000	29.56	2.050
60	3.620	6.881	277.0	1100	1175	1175	29.75	2.057
80	3.670	6.866	275.0	1025	1140	1120	27.68	2.024
100	3.725	6.886	280.5	1000	900	875	22.96	2.022

W/C .45

Water cured

0	3.677	6.881	263.3	910	925	940	23.27	1.925
5	3.648	6.879	268.0	1100	1025	1025	26.00	1.9766
10	3.647	6.884	272.0	1025	830	1075	26.63	2.004
20	3.657	6.877	278.7	835	800	100	22.14	2.0517

W/C .475

Water cured

0	3.617	6.871	265.3	925	925	625*	23.69	1.978
5	3.623	6.861	265.3	890	930	1050	24.51	1.9805
10	3.631	6.879	270.7	1030	975	1000	25.53	2.0059
20	3.645	6.883	276.0	1140	975	1000	26.33	2.035
80	3.680	6.879	280.3	920	875	1070	24.02	2.049

W/C - .465      Steam cured - 10 hrs. (Blended)

5	3.62	6.854	272.3	696	773	636	18.0	2.039
10	3.595	6.836	277.3	700	1145	709	22.05	2.102
20	3.57	6.840	280.0	1275	1000	1080	29.16	2.134

A.2.3 Portland (Interground) water curedGT - 4 hours

5	3.650	6.877	265.3	850	965	775*	21.89	1.957
10	3.694	6.873	272.3	1025	1075	1015	26.03	1.99

GT - 6 hrs. water cured (Interground)

5	3.655	6.864	267.0	960	875	1005	23.93	1.974
10	3.674	6.857	275.0	1245	1255	1150*	31.29	2.021

GT - 8 hrs. water cured (Interground)

5	3.641	6.845	270	1200	1200	1200	30.68	2.015
---	-------	-------	-----	------	------	------	-------	-------

10	3.667	6.866	276	1375	1300	1340	34.25	2.043
----	-------	-------	-----	------	------	------	-------	-------

A.2.4 1:4 lime/Ash ratio cement with portland (interground)GT - 4 hours

5	3.605	6.843	256	1000	965	950	24.945	1.9308
10	3.620	6.850	256.0	1175	1170	1000	28.774	1.9189

GT - 6 hrs.

5	3.611	6.847	257.7	1220	970	1200	29.085	1.9382
10	3.615	6.842	261.00	1150	1125	1150	29.396	1.9637

GT - 8 hrs.

5	3.60	6.850	261.7	1180	1220	1130	30.19	1.9731
10	3.652	6.871	267.3	1170	1190	1150	30.218	1.974

A.2.5 Ash ground for 8 hours with large cylinders and  
mixed with portland.

Ash	Port	H <sub>av</sub>	D <sub>av</sub>	M <sub>av</sub>	BL <sub>1</sub>	BL <sub>2</sub>	BL <sub>3</sub>	TS <sub>av</sub>	B.D <sub>av</sub>
25	75	3.668	6.883	282.7	1260	1285	1260	31.97	2.0713
50	50	3.659	6.852	285.00	1275	1300	1400	33.65	2.1123
75	25	3.633	6.885	270.00	750	800	700	19.10	1.9961

# A-2.6 HCl

Days	R.H.A. - PORT						PORTLAND							
	H	D	W	VOL	%	V	%	W	D	H	VOL	%	V	%
0	3.522	6.910	282.5	132.0795	-	-	-	3.610	6.920	289.0	135.7717	-	-	-
	3.622	6.964	291.6	137.9608	-	-	-	3.616	6.910	290.0	135.6046	-	-	-
10	3.498	6.878	276.0	129.96727	1.5991	2.30	3.580	6.786	277	277	129.47938	4.6345	4.152	4.152
	3.620	6.850	287.0	133.4073	3.3006	1.54	3.600	6.776	274	274	129.81927	4.2663	5.5172	5.5172
20	3.492	6.870	274	129.4427	1.9964	3.0088	3.572	6.758	273	273	128.12612	5.6312	5.536	5.536
	3.616	6.838	286	132.7934	3.7456	1.8868	3.588	6.742	270	270	128.09135	5.5406	6.8966	6.8966
30	3.490	6.866	273	129.21796	2.1665	3.3628	3.564	6.722	270	270	126.48079	6.8430	6.5744	6.5744
	3.612	6.832	285	132.41382	4.0207	2.2298	3.574	6.728	267	267	127.0622	6.2995	7.931	7.931
45	3.486	6.860	275	128.84438	2.4494	2.6549	3.582	6.694	271	271	126.06277	7.1609	6.2284	6.2284
	3.620	6.830	284	132.26303	4.12999	2.573	3.586	6.710	266	266	126.80757	7.2247	8.276	8.276
60	3.480	6.842	278	127.94851	3.1276	1.5929	3.557	6.674	277	277	124.47101	8.3233	4.152	4.152
	3.604	6.830	287	132.0432	4.2893	1.5437	3.572	6.644	267	267	123.83989	8.6757	7.931	7.931



## Acetic Acid

S	RHT - FORT				PORT											
	H	D	M	V	-	V%	-	W%	H	D	M	V	-	V%	-	W%
3.564	6.874	285.5	132.266	-	-	3.608	6.910	290.5	135.30458	-	-	-	-	-	-	-
3.590	6.896	291.5	134.085	-	-	3.588	6.880	289.0	133.38874	-	-	-	-	-	-	-
3.560	6.844	282.0	130.9664	0.98221	1.226	3.606	6.834	284.0	132.2713	2.242	2.24	2.242	2.24	2.24	2.24	2.24
5.590	6.872	289.0	133.1529	0.6948	0.8576	3.582	6.820	281.0	130.85315	1.900	2.768	1.900	2.768	2.768	2.768	2.768
3.554	6.840	280	130.593	1.2646	1.9264	3.602	6.816	281.0	131.4295	2.864	3.27	2.864	3.27	3.27	3.27	3.27
3.582	6.870	287.0	132.7789	0.9738	1.544	3.576	6.798	278.0	129.7925	2.696	3.806	2.696	3.806	3.806	3.806	3.806
3.542	6.838	279	130.0758	1.6555	2.2767	3.596	6.792	279.0	130.288	3.708	3.9587	3.708	3.9587	3.9587	3.9587	3.9587
3.576	6.866	285	132.4021	1.2550	2.2298	3.568	6.772	275.0	128.5135	3.655	4.844	3.655	4.844	4.844	4.844	4.844
3.534	6.888	279	129.78204	1.8777	2.2767	3.590	6.770	278	129.2295	4.49	4.303	4.49	4.303	4.303	4.303	4.303
3.568	6.870	285	132.2599	1.3611	2.2298	3.564	6.750	274	127.53668	4.387	5.19	4.387	5.19	5.19	5.19	5.19
3.550	6.840	279	130.4459	1.3758	2.2767	3.582	6.770	278	128.9415	4.783	4.303	4.783	4.303	4.303	4.303	4.303
3.594	6.850	287	132.4491	1.2200	1.544	3.560	6.730	274	126.6397	5.0597	5.19	5.0597	5.19	5.19	5.19	5.19

ium Sulphate

PORTLAND										ASHMOH				
H	D	W	Vol.	% V	%	W	H	D	W	Vol.	% V	%	V	W
3.632	6.850	293	133.84493	-	-	-	3.574	6.866	275	132.32808	-	-	-	-
3.666	6.878	297	136.20927	-	-	-	3.584	6.878	289	133.16258	-	-	-	-
3.636	6.856	296	134.2317	.29	1.02	1.02	3.574	6.860	277	+132.09691	-.175	.73		
3.672	6.880	300	136.51156	.22	1.01	1.01	3.570	6.872	290	-132.4111	-.565	.35		
3.638	6.866	299	134.6976	.633	2.04	2.04	3.506	6.850	280	131.41724	-.69	1.82		
3.674	6.892	305	137.06279	.627	2.02	2.02	3.558	6.864	293	131.65894	-1.13	1.38		
3.646	6.874	301	135.30866	1.09	2.73	2.73	3.562	6.848	282	131.19317	-.86	2.555		
3.678	6.914	305	138.0894	1.380	2.69	2.69	3.556	6.860	294	131.41162	-1.299	1.73		
3.650	6.900	302	136.48374	1.97	3.07	3.07	3.600	6.870	279	133.44609	.844	1.45		
3.680	6.914	306	138.16449	1.435	3.03	3.03	3.590	6.870	291	133.0754	-.065	.69		
3.680	6.926	303	138.6445	3.58	3.41	3.41	3.626	6.878	278	134.35153	1.529	1.09		
3.694	6.936	307	139.6546	2.539	3.37	3.37	3.612	6.882	290	134.35906	.8985	.35		

RHA - PORT										PORT			
H	D	W	V	% V	% W	H	D	W	V	%	V	%	
3.508	6.916	286.0	131.78301	-	-	3.586	6.904	287.5	134.24611	-	-	-	
3.516	6.914	282.5	132.00716	-	-	3.576	6.916	288.0	134.33753	-	-	-	
3.482	6.904	284.5	130.35275	-1.0853	-0.5245	3.562	6.886	287.0	132.65323	-1.1865	-0.1739	-	
3.506	6.868	281.5	129.886	-1.6068	-0.354	3.572	6.866	288.0	132.25403	-1.5509	-0.000	-	
3.476	6.900	282.00	129.97739	-1.370	-1.399	3.560	6.884	286.0	132.50174	-1.2994	-0.5217	-	
3.502	6.864	280.00	129.5867	-1.8336	-0.885	3.568	6.862	286.0	131.95205	-1.7757	-0.694	-	
3.747	6.894	284.00	129.6768	-1.5983	-0.6993	3.560	6.882	286.5	132.42474	-1.3567	-0.3478	-	
3.500	6.866	282.00	129.5882	-1.8324	-0.177	3.566	6.858	287.5	131.72438	-1.9452	-0.1736	-	
3.482	6.890	287.00	129.8246	-1.4861	+0.35	3.564	6.880	288.00	132.4965	-1.3032	+0.1739	-	
3.540	6.870	284.00	131.222	-0.5948	+0.531	3.566	6.856	289.00	131.64756	-2.0024	+0.3472	-	
3.500	6.890	288.00	130.4958	-0.7968	+0.6993	3.566	6.866	291.00	132.03288	-1.6494	+1.2174	-	
3.542	6.882	287.00	131.755	-0.191	+1.593	3.570	6.858	292.00	131.87214	-1.8352	+1.3889	-	

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SYS	RHA - PORT						PORT					
	H	D	W	V	V	W	H	D	W	V	V	+
3	3.522	6.872	286.0	130.63078	-	-	3.640	6.896	290.00	135.95205	-	-
	3.612	6.880	290.0	134.28097	-	-	3.604	6.872	290.00	133.67215	-	-
0	3.514	6.850	287.0	129.50089	-0.865	+0.3497	3.614	6.884	294.00	134.5116	-1.0595	+0.685
	3.564	6.864	293.0	131.88096	-1.7873	+1.0344	3.600	6.864	292.00	133.21309	-0.3434	+0.6897
0	3.510	6.846	285.50	129.20246	-1.0934	-0.17483	3.610	6.878	293.00	134.12861	-1.3412	+0.3425
	3.558	6.858	292.00	131.42887	-2.12398	+0.6897	3.594	6.868	291.00	133.14612	-0.3935	+0.3448
0	3.506	6.850	284.00	129.20607	-1.0906	-0.6993	3.608	6.876	292.00	133.9764	-1.4532	+0.0000
	3.550	6.860	290.00	131.20985	-2.2871	0.0000	3.590	6.872	290.00	133.1529	0.3884	+0.0000
50	3.508	6.852	282.00	129.35528	-0.9764	-1.3986	3.604	6.874	288.00	133.74997	-1.6197	-1.3699
	3.552	6.864	288.00	131.43692	-2.11798	-0.6897	3.598	6.868	285.00	133.29431	-0.2827	-1.724

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